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A MANUAL OF CHEMICAL PLANT

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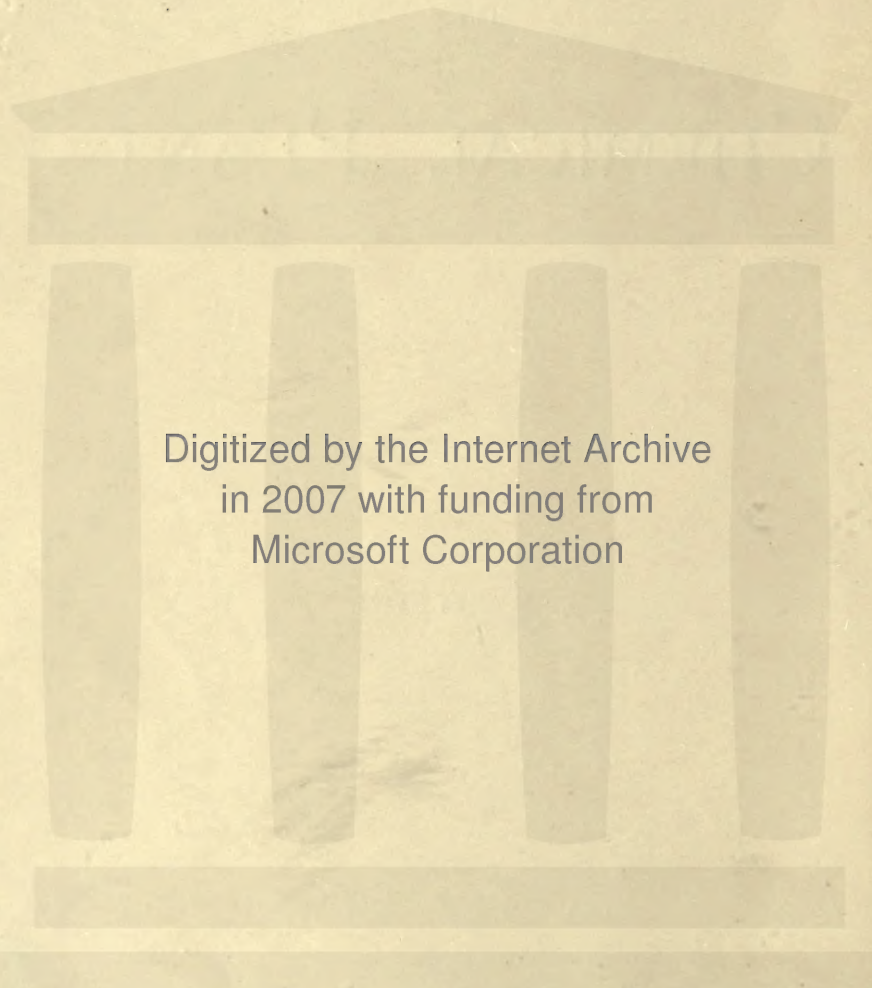
A MANUAL
OF
CHEMICAL PLANT

By
S. S. DYSON

Editor of "Chemical Engineering"

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Preface

Twenty years work amongst the literature of chemical technology has resulted in the accumulation by the author of a mass of data on chemical plant the value of which has been proved by constant testing on the part of chemical engineers and chemical manufacturers in the carrying out of process operations. Recently the author has been engaged in the arrangement of this matter, and a consideration of its varied nature, the carefulness of its selection, and the extent of its applications has led him to believe that a manual embodying such data would be found of practical value by technical chemists generally, and that the present juncture, promising as it does to be the beginning of a new era of development in the British chemical industry, is timely for the publication of such a manual.

It is not the intention of the author to write a treatise on Chemical Engineering—that has already been done by his late respected chief, Mr. George E. Davis, on whose staff he had the advantage and privilege of serving for some fifteen years. Rather is it his object to place in the hands of the chemical engineer, the chemical manufacturer, the chemical works manager, and the student of chemical technology the results of a long and patient examination of the claims of almost every new piece of chemical plant which has been introduced during the last quarter of a century. To this he has added an exhaustive analysis of the patent literature of the same period, and he believes that the resultant work will form an almost indispensable companion volume to the handbooks of chemical engineering and industrial chemistry already published. Moreover, it is hoped that the manual will also be found of service by works managers in the many industries where chemical plant is more or less employed for the production of chemicals necessary for process operations, for the recovery of by-products from such operations, or for the working up of the by-products after recovery.

It is no part of the author's plan, in compiling this manual, to enter into the question of the design of chemical works or the principles underlying the construction of chemical plant. That is the business of the chemical engineer, and for the theory of the subject the reader must be referred to the literature of chemical engineering. The task of the author is to present a record, at far greater length than is possible in any general chemical treatise, of the practical outcome of research and experiment as embodied in the range of plant which is actually available for the carrying out of the operations and processes of industrial chemistry, and the entire subject has been treated from this point of view. Especially is this the case with the patent literature which has been dealt with. The examination of some thousands of specifications of patents relating to chemical plant and processes has inevitably resulted in the laying down of very sharp lines of demarcation between the purely theoretical patentee and the inventor whose "improvements" reach the stage of a commercially successful apparatus or process. The author's work for the last twenty years has been the recording of progress in chemical technology from this standpoint, and the present volume represents the fruit of his labours so far as chemical plant is concerned.

The arrangement of the subject matter of the book does not follow any stereotyped lines. It is frankly one of convenience for presentation and publication in sectional form. Important group plant, such as ammonia recovery plant (including a lengthy section on by-product coking and the recovery of residuals), tar distilling plant, alkali plant generally, vitriol plant in particular, hydrochloric and nitric acid plant, explosives plant, cyanide recovery plant, wood distillation plant, etc., are treated in separate sections; other sections are devoted to the consideration at length of filtration plant, evaporating and concentrating apparatus, hydro-extractors, grinding and mixing machinery, pumping machinery, acid elevators, air-lifts, air and gas compressors, blowers, stoneware plant, weighing and measuring machinery, conveying and elevating plant, the measurement and regulation of temperature, etc. The generation of steam, gas, and electrical power has been treated at length, the various types of steam boilers, mechanical stokers, steam engines, steam turbines, etc., being fully described and illustrated. A special section is devoted to the gas producer, all the standard types being included. The filtration and purification of water for industrial purposes, and the treatment of effluents, are also the subject of a special section, and a chapter on miscellaneous general plant takes in a variety of apparatus which

does not fall into any particular group, the net result being a far more exhaustive and comprehensive study of the whole subject matter than is usually available in the pages of any chemical treatise.

The author is indebted to many of the principal and well-known engineering firms for generous assistance in dealing with special plant for which they have established reputation, and to the many chemical engineers and chemical manufacturers who have so readily subscribed for the book. The response to his preliminary prospectus has been so encouraging that he has entirely re-cast the lines of the work on a more comprehensive scale, and he believes that his manual will be found to place in the hands of those in executive positions much of the information which they require in deciding many of the problems inseparable from the selection of chemical plant and the economical working of the factories under their control.

A MANUAL OF CHEMICAL PLANT

CHAPTER I.

AMMONIA RECOVERY.

The recovery of ammonia in the destructive distillation of coal during the manufacture of coal gas, and from the carbonisation of coal in coke ovens, has developed very rapidly during recent years, and has now assumed the proportions of an important industry. In 1900, according to the Chief Inspector of Alkali Works, the quantity of ammonia recovered as a by-product from gas works, expressed in terms of ammonium sulphate, was 142,419 tons, and from coke ovens 10,393 tons; in 1915 the recovery had risen to 173,675 tons and 145,406 tons respectively, giving an aggregate of 319,081 tons out of a total production in the United Kingdom of 426,267 tons, the balance being recovered in iron works, shale works, from producer gas plants, and from bone and coal carbonising works, the total production having a value, at average peace prices, of well over £5,000,000.

The "Indirect" Method of Ammonia Recovery.—In gas works practice the old "indirect" method of ammonia recovery is still responsible for by far the greater part of the total production. As is well known, this method consists in cooling the hot gases from the retorts in the hydraulic main, where the ammoniacal liquor is condensed with the tar, the further cooling of the gases in the condensers, and their final

treatment with water in the scrubbers and washers, the ammoniacal liquor wells yielding a total volume of from 10 to 15 gallons per ton of coal carbonized.

The ammoniacal liquor is usually dealt with on the spot for the production of sulphate by distillation and absorption in sulphuric acid, or, much less frequently, for the production of liquid ammonia or ammonium chloride or nitrate. In gas works where no plant exists for the recovery of the ammonia, the liquor is either sold in its crude condition, or partially treated and disposed of in a concentrated form for subsequent working up elsewhere.

The Feldmann Still.—The Feldmann still (British Patent 3643 of 1882) is regarded as embodying the general principles upon which most of the continuous ammonia stills are constructed. The original Patent drawing is reproduced in Fig. 1.

The Feldmann apparatus consists of the column A (Fig. 1), situated above the decomposing vessel B, and of the column C arranged side by side with the latter. The ammoniacal water to be distilled passes from a tank or reservoir H, placed at a suitable height, through the tube *o*¹ into the vessel I. In this there is a ball cock by the action of which the level of the liquor in I is maintained at a constantly uniform height, a constantly uniform introduction of the ammoniacal water into the column being thus attained. From the vessel I the ammoniacal water passes through the tube *o* into the feed heater G, constructed on the principle of counter currents; in this it passes through a coiled pipe from below upwards, and then passes by the tube *a* into the upper compartment of the column A. From here the liquor passes through overflow pipes *u u* in the bottoms of the several compartments of the column A, downwards through a longer overflow pipe into the decomposing vessel B. Into this a force pump P pumps at suitable intervals through the tube *c* an appropriate quantity of milk of lime. The tube *c* opens into the exhaust tube of the vessel B, the tube being provided with a stop cock *z*. In the upper part of the decomposing vessel B underneath the lower division of the column A a steam pipe Z opens which conducts steam from the column C to the column A. In his specification Feldmann says:—"To induce complete intermingling of the ammoniacal water with the lime and to cause decomposition of the former by the latter, a steam-recoil wheel is provided in the lower part of the decomposing vessel B, into which wheel steam can be driven from the top by a special steam pipe *r*. The action is based on the principle of Segner's waterwheel or Barker's mill. To enable the lime stirred up by the steam wheel to settle more easily in the upper part of the vessel B, a seive bottom is provided

at about two-thirds of its height so as to impede the movement of the liquid in this part. The admission of steam to the recoil wheel is so regulated that the decomposed ammoniacal liquor flows off clear from the decomposing vessel B through the overflow tube *e* into the column *c*. By this means a more regular re-utilization of the excess of lime is also rendered possible. The steam is admitted into the lower part of the column *c* through the tube *h*. The supply is regulated by the adjust-

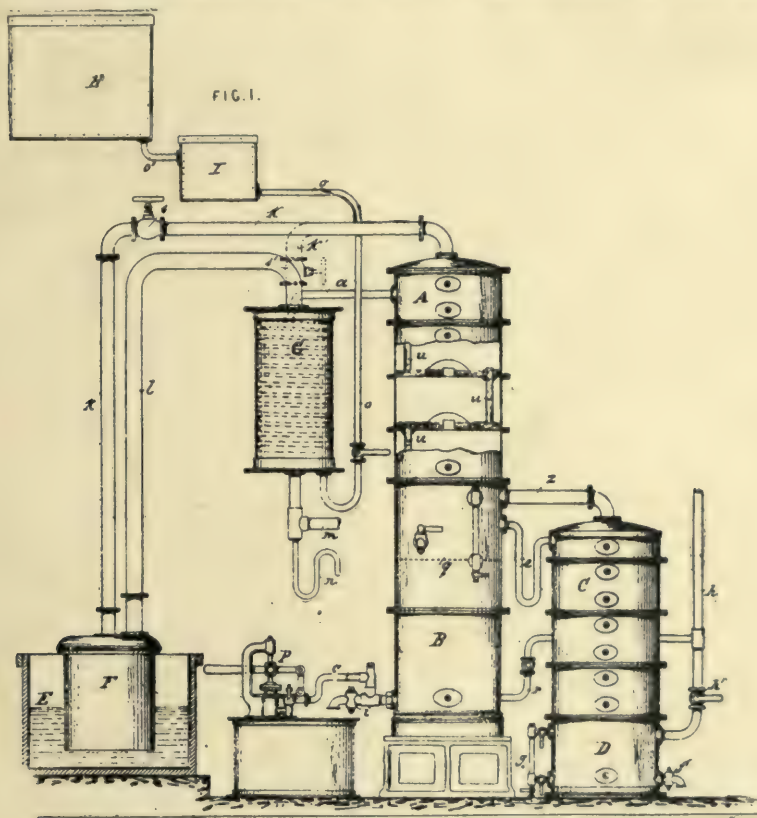


FIG. 1.—FELDMANN'S AMMONIA STILL.

ment of the cock *h*¹ according to the quantity and the strength of the admitted ammoniacal water so that a distillate of even strength may be obtained in all cases."

When the decomposed clear ammoniacal liquor has passed from top to bottom through all the compartments of the column *C* by means of overflow pipes arranged in the bottoms of the several compartments, it arrives completely separated into the lowest compartment *D* of the column *C*, whence it can flow off by a cock *f*. This cock can be properly adjusted by means of a level indicator *g* placed in the bottom compart-

ment D. The arrangement may be such that the decomposing vessel B with the column A are placed on the column C, in which case the steam from the top compartment of column C is led away sideways.

From the upper part of the column A a tube *k* leads to the saturator E, the waste gases passing through the pipe *l* into the feed-heater G, and thence, freed from aqueous vapour through a tube *m* leading from the bottom of the feed-heater, under the grate of a fire, whilst the condensed water is led away through *n*. If the apparatus is to serve for producing concentrated gas water, the steam discharge pipe *k* is directly connected with the feed heater G as indicated in dotted lines at *k*¹. In this case the valve *s* communicating with the box E is closed, and the valve *s*¹ provided in the branch tube *k* is opened; the tube *n* is then dispensed with. The outlet *m* at the bottom of the ammoniacal liquor is then connected with a small cooling device, as with strong ammoniacal liquors the cooling effected by the fresh supply of ammoniacal water is insufficient.

The Wilton Sulphate of Ammonia Plant.—A typical installation of the Wilton sulphate plant, as installed by the Chemical Engineering Co. and Wilton's Patent Furnace Co., of Hendon, London, N.W., is shown in Fig. 2.

Wilton, in his Complete Specification to British Patent 24,832 of 1901, observes that apparatus for the distillation of ammoniacal liquors as commonly used consists of superposed circular trays or chambers constituting a tower, each tray or chamber being provided with a pipe extending for a suitable distance above and below the bottom of the tray, the upper end of this pipe passing up to a suitable height above its tray to ensure a proper liquid level, and the lower extremity reaching down to approximately the liquid level of the tray next below. Each of these trays has a central opening to allow the steam by which the distillation is effected to rise up through the series of trays. Over the steam orifice of each tray is mounted a circular hood or baffle, usually serrated at its periphery, and which dips below the level of the liquid in the tray. The ammoniacal liquor passes down by the pipes through the series of trays, and the steam rises through the trays, bubbling out under the edges of the baffles through the liquor, and thence rising to the next tray or chamber. Apparatus constructed in this manner is very difficult to clean, even by the mudholes which are provided in the sides of the trays for the purpose, because the chambers or trays are necessarily shallow, and the pipes by which the liquor passes down from tray to tray extend up and down as mentioned to the liquid level in each tray. It is thus practically impossible to get at the interior of these

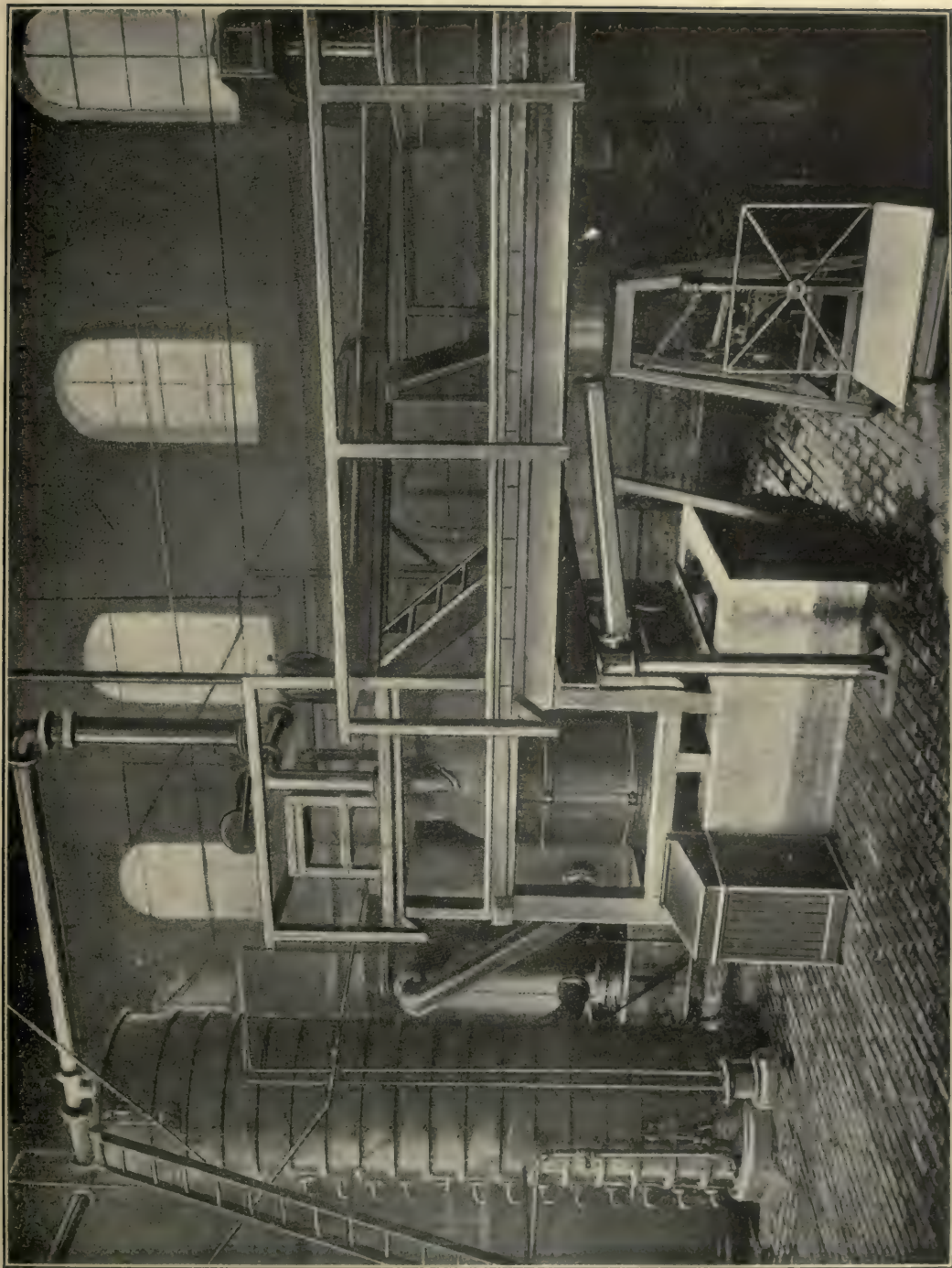


FIG. 2.—WILTON SULPHATE OF AMMONIA PLANT

pipes to clean them through the mudholes. Moreover, the hoods over the steam openings are too large to be removed through the mudholes, and consequently they cannot be cleaned with any degree of efficiency. Further, as the baffles are circular, and a considerable space exists

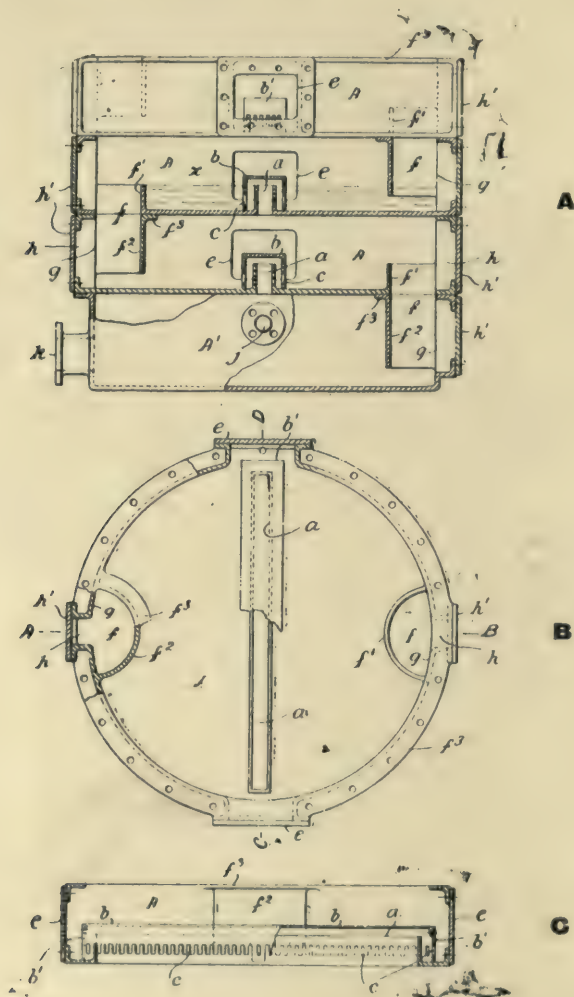


FIG. 3.—DETAILS OF WILTON STILL.

between their outer circumference and the sides of the trays or chambers, a large amount of liquor flows around the baffle to the outlet of the tray without coming under the action of the steam.

In the Wilton still as described in the Specification mentioned,

Fig. 3 B is a plan looking down upon a tray which is shown partly in section; A is a sectional elevation on line A—B of B, and showing three trays mounted upon a suitable base; and C is a section of one tray on line C—D, Fig. 3 B. The steam openings *a* of the trays or chambers A and the baffles or hoods *b* which depend into the liquor in the trays are made of somewhat narrow elongated form, and extend across the full width or nearly the full width of the trays. The bottom edge *c* of the baffles may be serrated as shown, or otherwise formed in the usual or other convenient way to facilitate the bubbling of the steam out from below the baffles and up through the liquor, the level of which is indicated at *x*. The inlet and outlet openings *f f* for the liquor to and from each tray are placed at opposite sides of the baffle *b*, which baffle thus constitutes a kind of bridge through or over which the liquor is compelled to pass in its passage through the tray from the inlet and outlet *f f*. The entire mass of liquor passing through the tray is thus brought into intimate and thorough contact with the steam which passes through the baffles.

In order that the apparatus may be readily accessible for cleaning or other purposes a mudhole *e* is provided in the wall of the tray A opposite each end of the elongated hoods *b* above described; through the mudholes *e* the hoods *b* can be removed when desired. The hoods are made with removable ends *b*¹ adapted to be bolted on or removed after the hood has been slid over, or before it is removed from the upwardly projecting steam pipe *a*. Further, with the same object of rendering the apparatus easy of access for cleaning or other purposes, the inlet and outlet pipes for the liquor from tray to tray are formed as vertical channels or troughs *f* of half round or other convenient section arranged directly against the side walls of the trays A so that the side walls, for instance the portions *g*, form part of the walls of the inlet and outlet pipes *f*. Mudholes *h* are also provided opening directly into the pipes or channels, so that by removing the mudhole covers *h*¹ the connecting pipes or channels *f* can be readily cleansed. The upper part *f*¹ of the pipes which is attached to the floor of the chambers or trays may be formed in one as shown, or fastened to it. This is also the case with the lower portion *f*², or it may be made as in the drawings, where the upper part *f*¹ is made in one with one tray, and the lower part *f*² cast upon the side of the tray beneath in this case. Suitable flanges *f*³ are provided for making the necessary joint. In Fig. 3A, A is the lower chamber of the tower fitted with the usual steam inlet and liquid outlet pipes *j k*, and formed with a part *f*² of a channel *f*, so as to make the required connection with the tray next above it.

The advantages claimed for apparatus constructed as above are

that it will operate with increased efficiency above that obtained by the old form of circular baffles, due to the fact that practically none of the

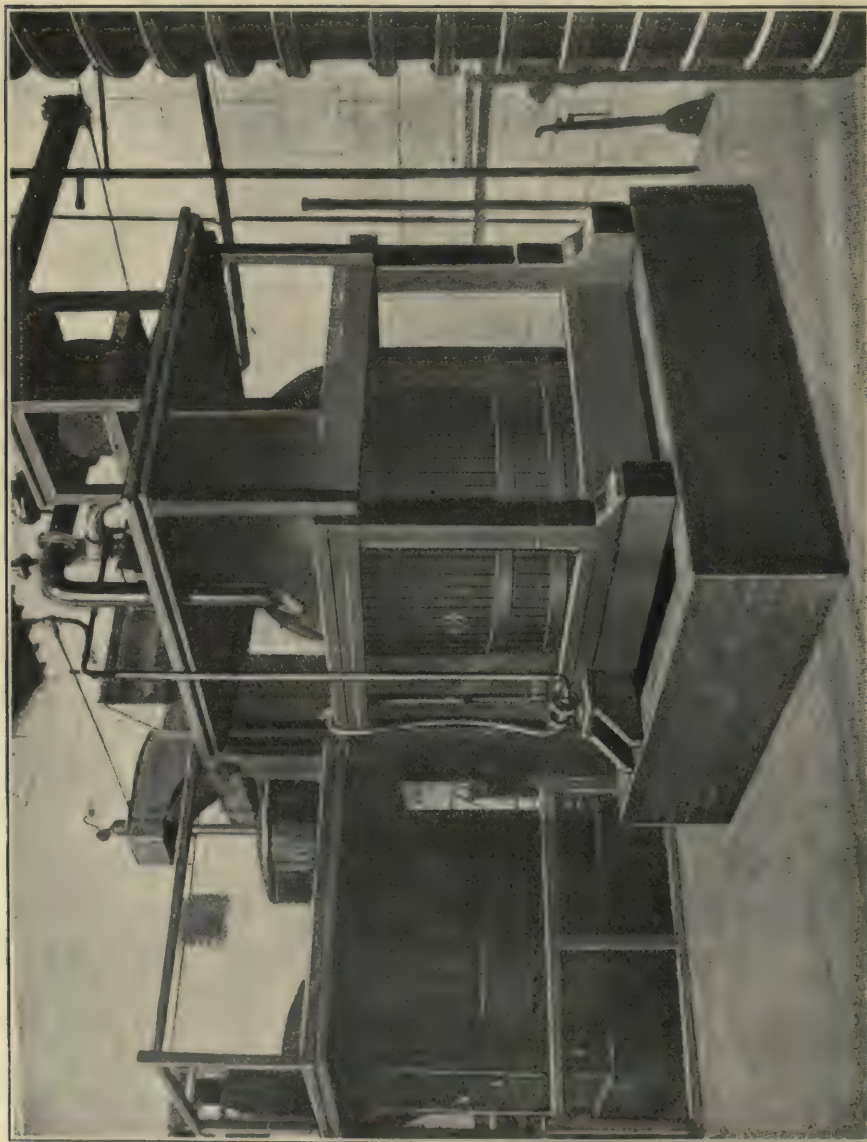


FIG. 4.—WILTON ENCLOSED SATURATOR.

liquor under treatment in passing through the trays can flow from the inlet to the outlet without passing under the influence of the steam issuing from the baffles; further, the number of the baffles may be increased with a corresponding increase in the area over which the steam

comes into contact with the liquor. When it is desired to clean the working parts, all that is necessary is to remove the covers of the mud-holes and slide out the hoods. The liquor channels also can be readily cleaned through the mudholes opening into them, and the hood, after cleaning, can be slid back into place, and the steam channel can also be cleaned from the mudholes, which allow of the removal of the hood.

An important feature of the Wilton sulphate plant is the patent saturator (Fig. 4). Fig. 5 is a reproduction of the drawing filed with Wilton's British Patent 5,205 of 1890.

A and B (Fig. 5) are vertical sections through the centre of saturators constructed according to this method. A vessel S is perfectly closed

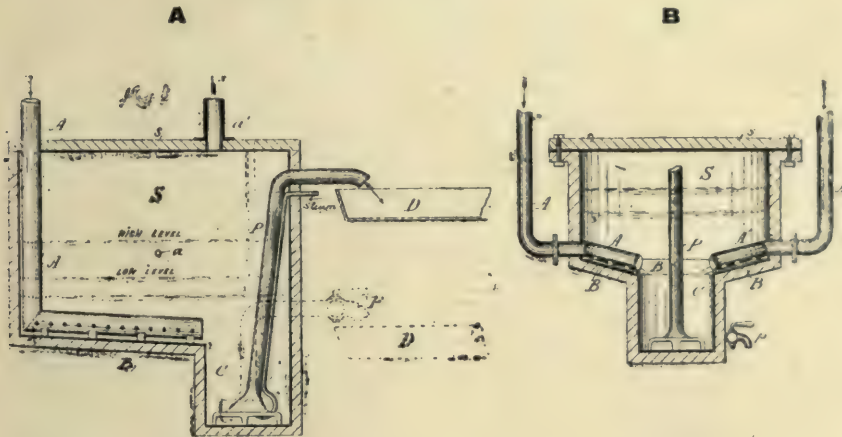


FIG 5.—DETAILS OF WILTON SATURATOR.

by a cover *s*, all of suitable strength and materials. The vessel may be square or rectangular as indicated, or it may be of circular form. Into this vessel the ammonia supply pipes *A* are led, and the discharge is arranged at such a level that a sufficient cubical space (in proportion to the capacity of any manufacturing plant) intervenes between the level of the discharge and the lowest part of the vessel. For this purpose it is convenient to make the saturator of an inverted **7** or **T** shape as illustrated, in which the vertical member forms a depositing chamber or well *C*, towards which the base *B* of the upper part may with advantage be inclined. In large saturators there may be two or more such depositing chambers. The ammonia pipe or pipes *A A* inside the saturator may be either straight or curved according to circumstances, and made to follow the inclines of the base *B*, and provided with the usual dis-

charging orifices; the acid pipe *a* and the escape pipe *a*¹ for waste gases may be arranged at any convenient points. The object of this arrangement is to ensure the circulation of more or less clear liquid in the neighbourhood of the ammonia discharge, while the deposit will all take place below and clear of this, and thus prevent the clogging of the ammonia discharge orifices. The capacity of the depositing chamber or chambers *C* must be sufficient to receive and contain all the salts that are deposited—it may bear a proportion to that of the vessel *S* (roughly) of about 1 to 3.

The salts deposited in the chamber *C* may be discharged by gravity through an ordinary blow-off pipe and tap

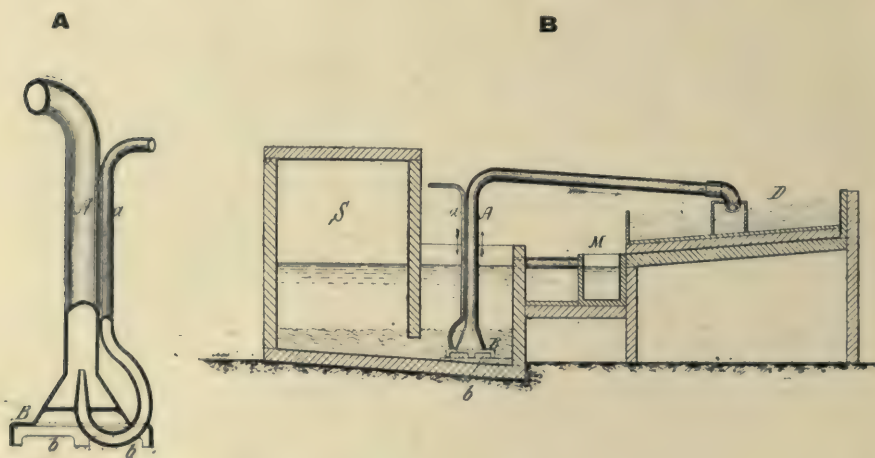


FIG. 6.—WILTON'S AUTOMATIC DISCHARGER.

placed at or near the base as indicated at *p*; by this arrangement, however, a certain quantity of the "mother liquor" is discharged with the salts which has to be raised and returned to the saturator. To obviate the raising it is better to lift and discharge the salts at a higher level, say about two feet above the general level of the acid, so that any liquor can drain back into the saturator by gravity. This can be efficiently accomplished by the use of the automatic discharger arranged as shown at *P*, and discharging into drainer *D*.

By the above-described arrangements it is claimed that one saturator will suffice for four open trough saturators of the same capacity and constructed in the usual manner, for the following reasons: The vessel *S* being perfectly closed, no splashing over of the liquids can

take place, neither can the noxious fumes escape into the atmosphere about the saturator; nor need any stoppage or change take place, and thus the process is continuous; further, no manual labour is required

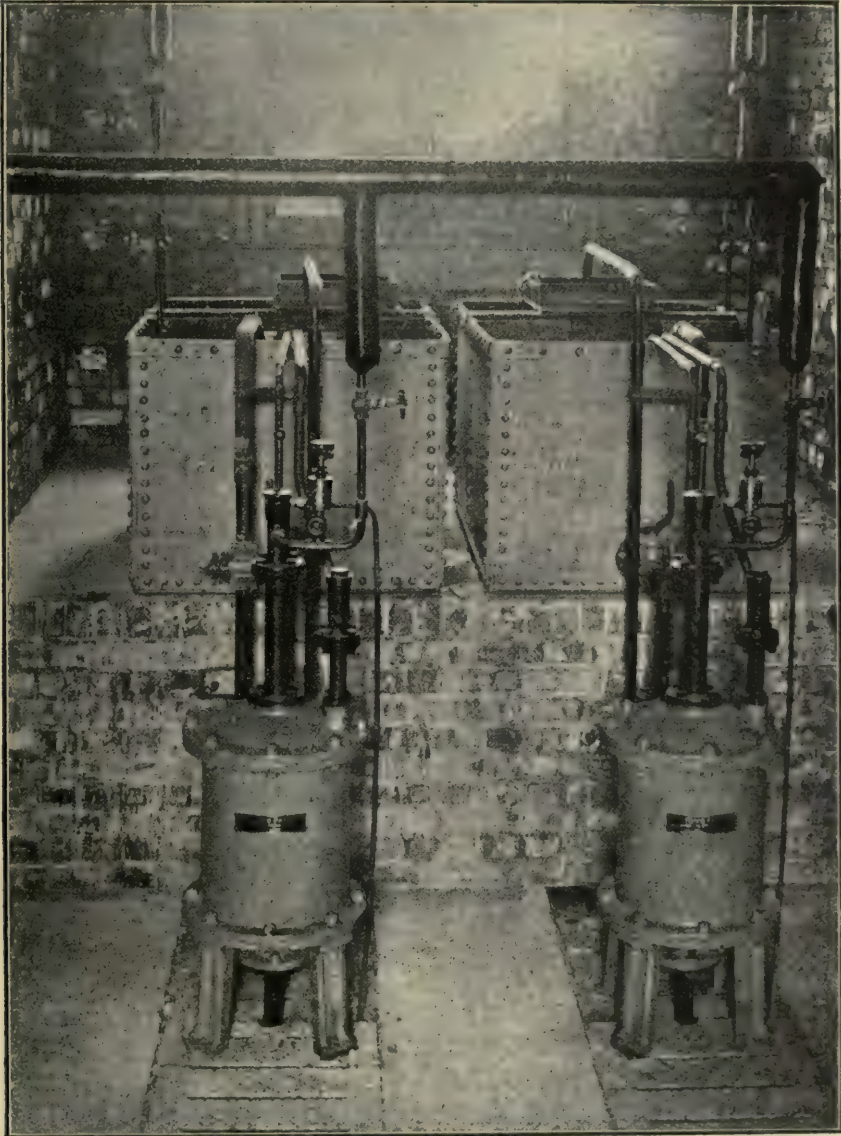


FIG. 7.—WILTON LIMING APPARATUS.

either during the manufacture or the discharge of the sulphate of ammonia. It will be obvious that in practice a similar result might be obtained by leaving the top parts of the front of the vessel S open so

as to form an open trough, and applying the usual fixed curtain, but there is no advantage in this method of construction.

The automatic discharger referred to above is shown in detail in Fig. 6, in which A is an elevation partly in section of an ejector, and B is a section through an ordinary arrangement of saturator, mother liquor trough and drainer, showing the ejector fixed in a depositing vessel, which in this case is the saturator itself. (British Patent 9,225 of 1889.)

A is the ejector made of lead or other metal adapted to withstand the action of the liquids employed; it consists of a pipe or duct which may advantageously be constructed as illustrated, with a bell or trumpet-shaped lower end terminating in or at a base plate B, with slots or passages *b b* for the ingress of deposited salts. To this is applied a steam pipe *a* provided with the usual cock, and terminating in a nozzle at or near the base of the ejector A.

An ejector constructed in this manner is placed and fixed in the depositing vessel—whether it be the saturator itself or a depositing vessel (or “ejector tank”) in connection with a saturator. In Fig. 6 B the ejector is fixed in the trough of an ordinary saturator S, from which sulphate of ammonia is usually removed by hand, but the ejector may be applied to any construction or type of saturator. M is the mother liquor trough, D the drainer. The upper end of ejector A is connected by an ordinary lead pipe to a point of support above the drainer D which conveys the discharge of the ejector to the drainer, which may have a midfeather to limit the flow of deposit, and has an overflow for the excess liquor.

On turning on steam the discharge of salts from the ejector tank to the drainer commences, and continues without any manual assistance until steam is turned off; the pressure of steam is regulated by the steam cock, and a small pressure only is required.

A natural circulation may be used, or to augment the carrying power of the liquid agent employed when there are many connected saturators, a current may be induced through the whole series by a steam jet or other suitable means.

The Wilton liming apparatus is shown in Fig. 7. For this the makers claim extreme simplicity and low working costs. The apparatus consists of a rectangular wrought-iron tank, which is divided into two compartments, so that the actual milk of lime will overflow from one to the other and feed the pumps, while the sludge remains in the first compartment. The lime is placed in wire baskets and slaked with the spent liquor from the still; from the tanks it runs by gravitation to the

pumps, whence it is forced at boiling temperature into the mixer.

The lime pump is very simple, and has no working parts. The

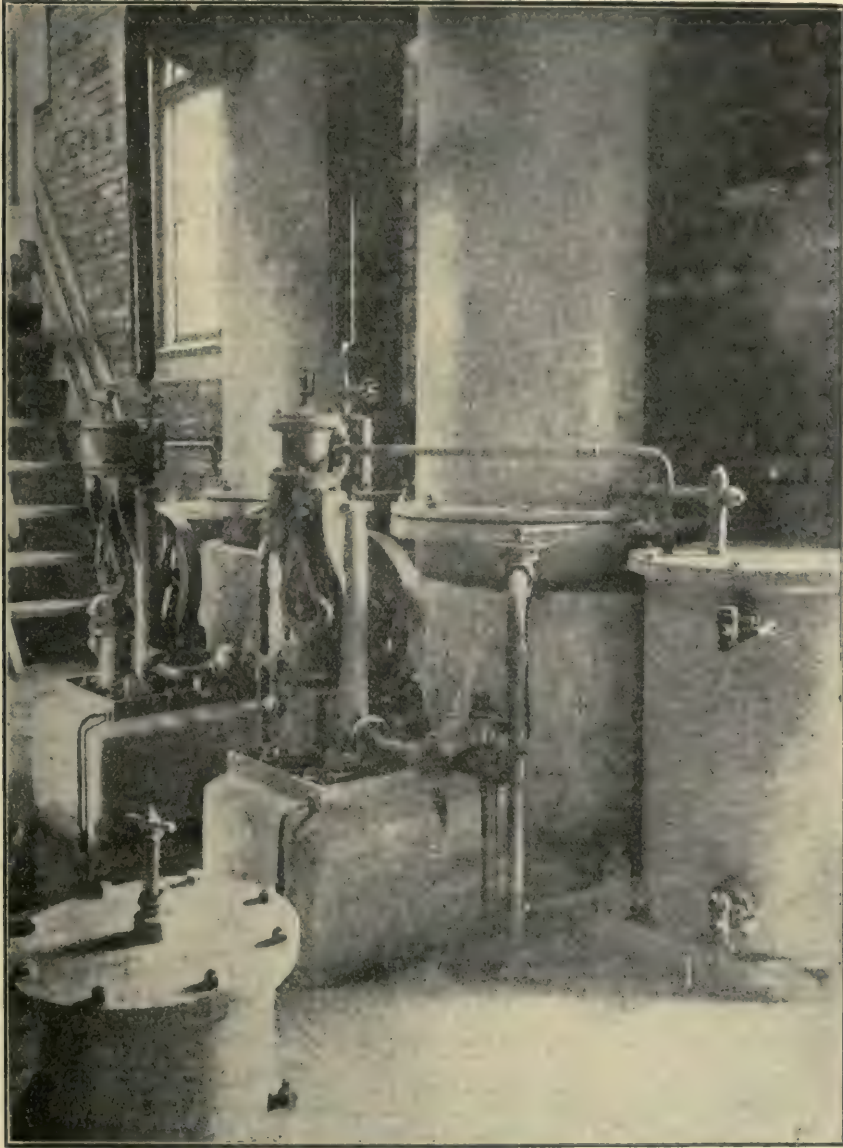


FIG. 8.—WILTON DEVIL LIQUOR APPARATUS.

milk of lime, when filling the pump body, causes a steel float to rise, which works an automatic valve. When the pump is full this valve shuts off the supply and admits steam, which forces the contents to the

mixer; the float in falling shuts off the steam and re-opens the milk of lime supply, when the operation is repeated. The pump can be adjusted to any required rate, the whole operation being automatic.

Fig. 8 shows the Wilton apparatus for dealing with the devil liquor. The apparatus consists of a cast-iron closed receiver and suitable ram type pump, so that the liquor can be pumped with the ammoniacal liquor to the still. On small works it is usual to run this liquor away with the waste liquor, or else return it to the liquor well, but the latter method involves the risk of blockage, as it contains a large percentage of naphthalene salts.

The Dempster Sulphate of Ammonia Plant.—The sulphate of ammonia plant of Messrs. R. and J. Dempster, Ltd., Oldham Road, Manchester, is shown in Fig. 9.

The still is divided into primary and secondary portions, the first for the liberation of the free ammonia, the second for the extraction of the fixed ammonia. Various improvements have been made in the construction of the still, notably in the method of accessibility to the interior for cleaning. Both the primary and secondary stills are so designed that the whole of one side (and in the case of very large stills, both sides) is made entirely removable in small plates, and access to the inside gained without disturbing any of the connections to or from the stills, thus enabling the whole of the interior to be thoroughly and efficiently cleaned. In addition to this, handholes are fixed adjacent to the overflows in each tray, so that should the still show signs of priming, the removal of these small hand-hole covers enables the overflows to be thoroughly examined, and cleaned from the outside. The overflows are suitably sealed, and made of very large area, to prevent blockage or back pressure.

The trays consist of a series of bubbling lutes, each provided with a serrated hood, securely held down in its correct position by means of adjustable bearer bars, thus preventing the possibility of the hoods being disturbed, and ensuring them being fixed in their correct position. Each tray is also provided with a draining plug, so that any one, or the whole of the trays, can be emptied.

Between the primary and secondary still a lime interceptor fitted with a flushing cock is provided. This lime interceptor prevents any lumps of lime being carried forward to the secondary still, and is provided with a removable cover, so that the interior can be thoroughly cleaned. The flushing cock is connected to the pipe leading to the lime settling tank, so that the waste lime is properly recovered. The sludge and overflow pipes from the stills are provided with crosses and plugs for cleaning purposes. The lime mixer in the bottom

of the primary still is on the makers' own principle, worked by means of a steam injector. The lime inlet pipe is carried into the still,

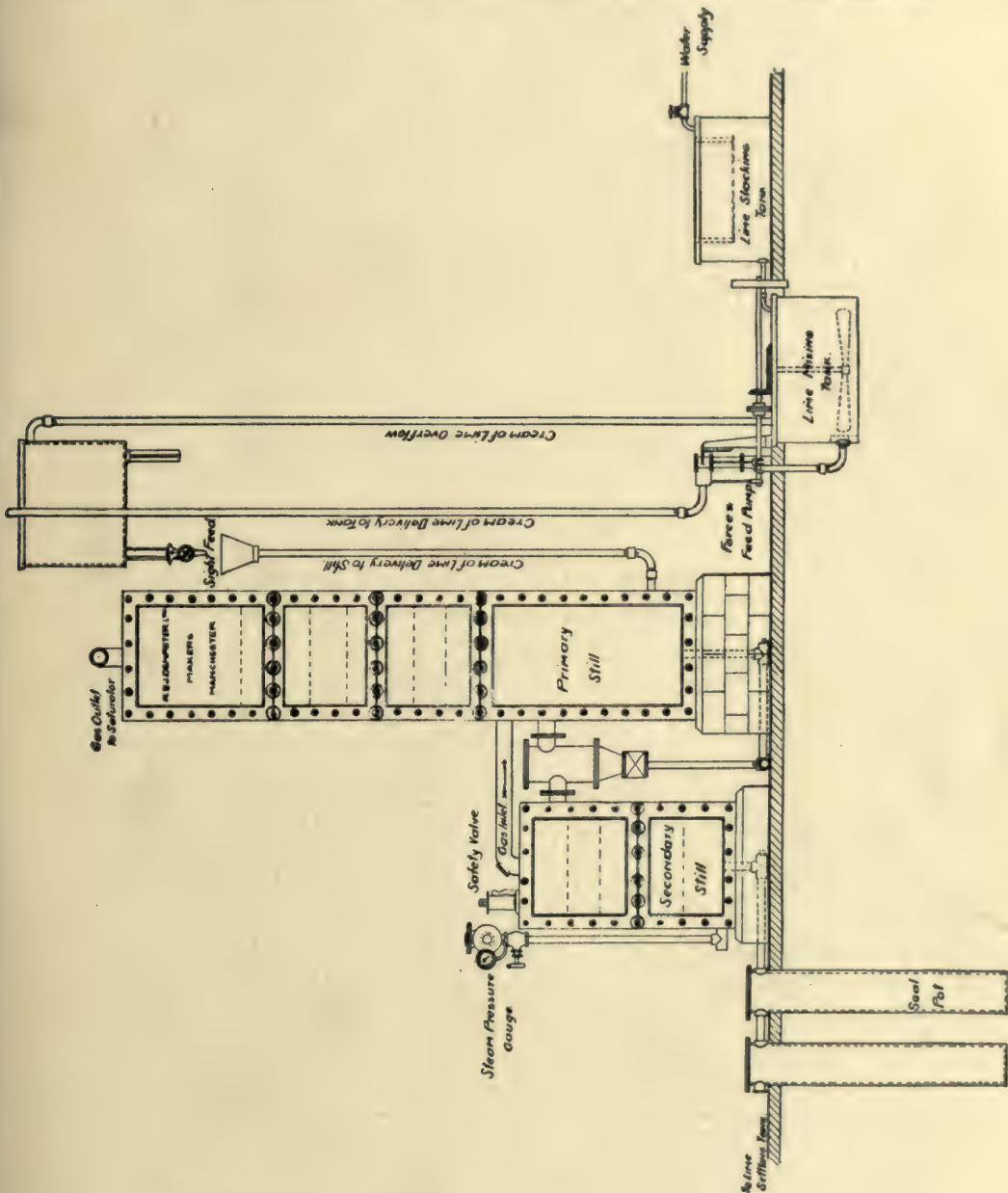


FIG. 9.—SULPHATE OF AMMONIA PLANT BY R. & J. DEMPSTER, LTD.

and delivers the cream of lime immediately opposite the injector, which forces the lime up the mixer, and thoroughly circulates it with the

liquor, leaving the primary still for final steaming in the secondary still.

The steam pipes to the still have ample area to ensure an adequate supply of steam, the required pressure being obtained by means of a reducing valve, and in order to prevent any excess of pressure being generated in the still, safety valves are fixed on the secondary still, and on the liming chamber of the primary still.

Ample space is allowed on top of the primary still to prevent any chance of the liquor backing up the ammonia gas pipe, and thus getting into the saturator.

The Davis Sulphate of Ammonia Still.—The Davis still (British Patent 15,540 of 1887) is shown in Fig. 10. The still consists of a metal cylinder as shown by A B, and is fitted with a number of trays shown at C. Each tray is fitted with an overflow pipe D dipping into a shallow well E, and these trays are perforated with a number of small holes as shown in the plan P, through which the vapours pass when the still is at work. About one hole, three-sixteenths of an inch in diameter, in every square inch of tray is the number found to be sufficient. Any volatile ammonia compound present in the solution undergoing distillation is driven off in the upper portion of the column or still A, and the partly freed liquor passing through the pipe G into the lime vessel or box K becomes mixed with the lime or soda introduced through the pipe M. From K the liquor finds its way into the column again through the pipe H, and in passing down the column over the perforated trays becomes denuded of its fixed ammonia, finding egress from the column at the outlet I by means of a series of one or more U tubes which allows of the escape of liquor and still retains the steam.

In practice the liquor to be distilled is run into the still or column—preferably having first been heated by means of the waste steam—by means of the smaller pipe on the top of the still, and when steam is admitted by the pipe N the liquor will form a layer on each tray of a depth corresponding with the top of the overflow pipes D, by means of which it runs from tray to tray until it finally reaches the point I, almost entirely freed from its ammonia.

The steam and other vapours pass through the holes in the trays, and find their way through the liquor standing on each into the spaces J above, and so on from tray to tray until they finally escape, highly charged with ammonia, by the large pipe upon the top of the column.

When the steam is turned off the liquor falls from tray to tray, and any tar which may have settled on these finds its way by the pipe G into the lime box K, from whence it is removed, together with the refuse

lime, by means of the tap L, or by the manhole with which this box is provided.

The inventor observes: "This form of apparatus already described answers best when used for the distillation of the quantity of liquor which it is calculated to work. When the quantity of liquor to be

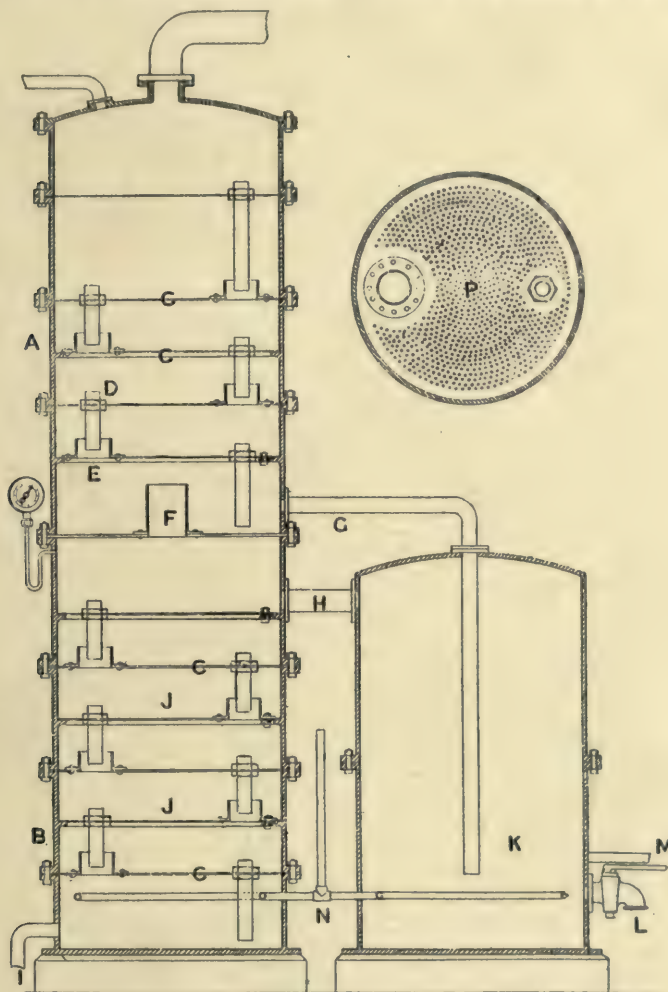


FIG. 10.—DAVIS AMMONIA STILL.

distilled is very irregular I prefer to make the perforations in the top of a flat cylindrical cap shown at A in Fig. 11. At B in the same drawing is shown one of these caps *in situ*, while the plan C exhibits the method of perforation. These perforated caps and trays may then replace the simple perforated trays, . . . I do not confine myself to the practice of placing the lime vessel K in the exact situation shown with the

drawing, as it is part of my invention to be able to vary the situation aforesaid according to the quality of the ammonia it is intended to produce. Thus, if a very caustic ammonia be required the lime vessel K must be placed nearer to the still top than is indicated in the drawing."

According to a later Patent (16,349 of 1888, Fig. 12, A, B, and C) the caps covering the steam necks are made of such a form that the outer edge in plan is represented by a deep sinuous curve, as shown at A, and the whole of this contour line is serrated to allow of the escape of

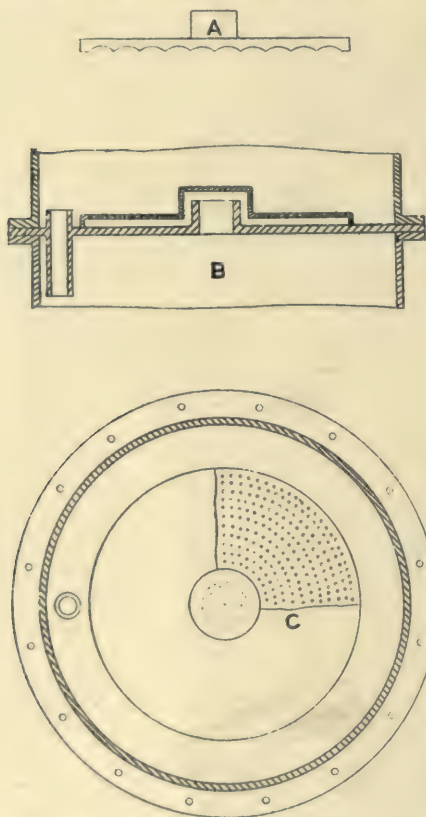


FIG. 11.—TRAYS OF DAVIS AMMONIA STILL.

the steam and gases from underneath. A section of one of these caps is shown at B, Fig. 12, covering the short neck *a*. Stops S are also cast upon the trays or plates upon which the caps A rest as shown at A, in order to compel the liquid to travel the sinuous path from the inlet to the outlet of each tray or plate *b*.

The still, as described in the Patent last mentioned, is constructed in one column M in which a certain number of the trays and caps (preferably six) are employed for liberating the volatile ammonia; these trays and caps are placed at the top or upper part of the still. A sufficient number of similar trays and caps (also preferably six) are placed at the bottom of the column M for liberating the

fixed ammonia after it has been displaced from its acid combinations by means of lime. Between these two series of trays and caps is placed the liming vessel L, through which the steam and gases are conducted from the lower tray by means of the pipe B of wide bore, thus heating the milk of lime on its passage through it, the pipe C bringing the liquor into the liming chamber L from the trays above and the overflow pipe D conducting the liquor from the liming chamber into the trays below. The milk of lime is forced into the liming chamber at the pipe

G by any well-known means such as by the aid of a hand-pump, steam-pump or injector, and the lime is further heated and kept in agitation by the fixed steam pipe E.

In certain cases, as when the ammoniacal liquid to be distilled contains but little free ammonia, the lime chamber L is placed much nearer to the top of the still, so as to have a fewer number of trays

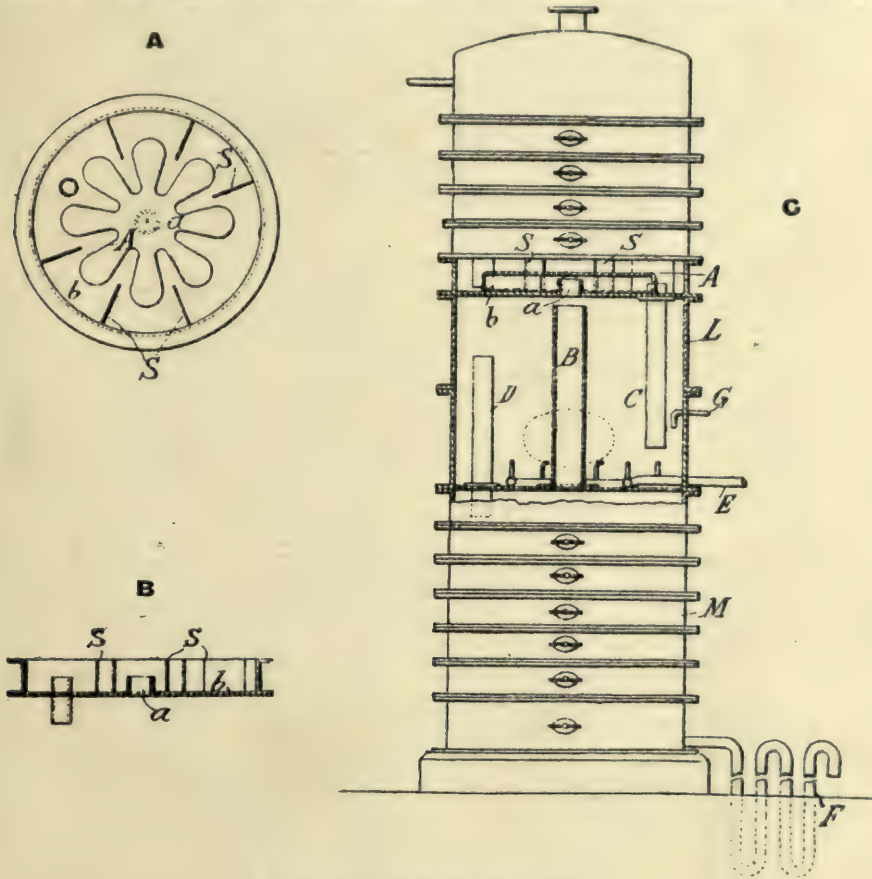


FIG. 12.—MODIFIED DAVIS STILL.

above it and more below it, while in some other cases, as when alkali waste is to be used in the place of lime, the liming chamber may be placed with all the trays below it.

A further modification (British Patent 26,996 of 1903) in the Davis still is shown in Fig. 13, in which the serrated caps covering each steam neck are fixed to the plate forming the tops of the chambers in which they rest, so that the serrations hang free in the liquid contained in the chamber, and therefore a small rake or special cleaner may be inserted underneath the caps in order to withdraw the lime deposit.

Four methods are shown by which the serrated caps may be fixed to the ceiling of the chambers, but it is always necessary to secure full submergence of the serrations of the cap, to allow a free flow for the liquor, and to secure that the gases and vapours passing from chamber to chamber find a ready passage over the serrated caps. A shows a method of securing the caps by means of a number of ordinary set screws which are screwed on the under side of the tray. In B the

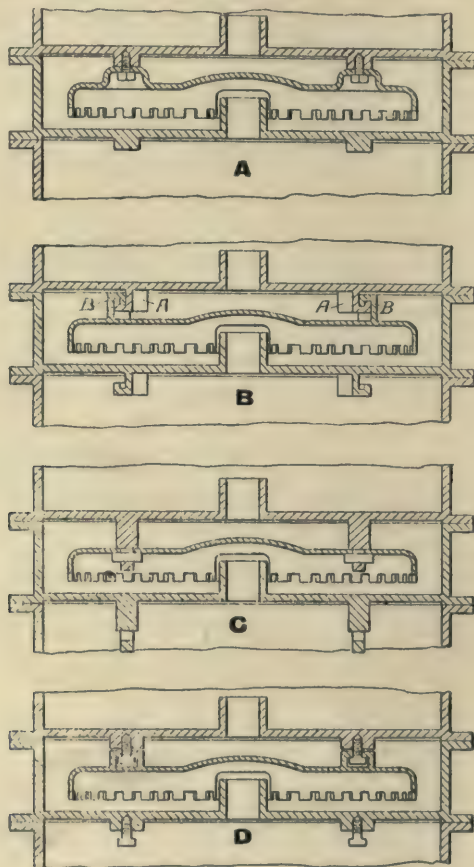


FIG 13.—METHODS OF FIXING CAPS OF TRAYS IN DAVIS STILL.

caps are secured by means of the fastening known as the "bayonet catch," consisting of a circular claw A which is cast on the bottom of the tray, and engages with a similar claw B cast on the top of the serrated cap. Several pairs of these claws are placed on the same circle, and the serrated cap is secured by elevating it until its claws are in the same plane as those on the tray, and then twisting the cap, when the claws become engaged as shown. C shows a method of securing the serrated cap by means of a cotter and bolt, the bolt being part of the

tray. D shows another method, in which a tee-headed set screw is made to engage in a corresponding horizontal slot on the top of the serrated cap, by twisting the cap in the same manner as in B.



FIG. 14.—AMMONIA STILL BY W. C. HOLMES & CO., LTD.

The Holmes Sulphate of Ammonia Plant.—Fig. 14 shows the type of sulphate still made by Messrs. W. C. Holmes and Co., Ltd., of the Wiltshire Iron Works, Huddersfield. The still consists of a column containing fixed and free portions, with liming chamber in the centre for treating, with cream of lime, the liquor which has already been passed

through the upper portion in which the free ammonia has been removed.

The design of the still aims at reducing to a minimum the liability of



FIG 15.—SMALL CIRCULAR STILL BY W. C. HOLMES & CO., LTD., SHOWING OVERFLOWS WITH DOORS REMOVED.

blockage and consequent frequent cleaning. Only one overflow duct is provided instead of two or more as in some other stills, but this is arranged

external to the still casing, and is of such ample dimensions that there is no constriction at the point of leaving or entering the still tray. These overflows are fitted with a cover the full width of the channel; this cover

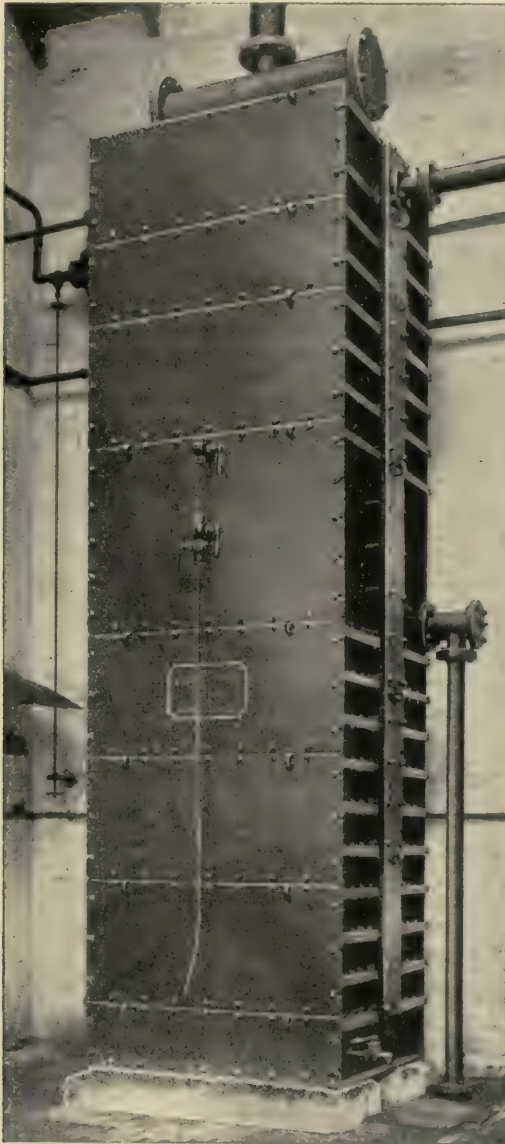


FIG. 16—SQUARE STILL BY W. C. HOLMES & CO., LTD., SHOWING CLEANING-OUT DOORS ON SIDE, AND OVERFLOWS.

may be easily removed, giving access to those portions of the still where lime deposit is likely to be found. The removal of the larger covers of

the still is thus only very occasionally necessary. If these covers are removed the bubbling hoods themselves may be also taken out, and the still stripped without dismantling the trays, as shown in Fig. 17.

Although the makers prefer the still in one column, as less steam is required, and there is less liability of blockage, the apparatus is made with two columns for erection where room does not allow for a single column. As the efficiency of all ammonia stills depends upon the actual contact between the descending liquor and the serrated edges of the hoods on each tray which form the bubbling surface between the ascending steam and gases and the descending liquor, a very high efficiency is claimed for this type, it being possible to reduce the spent liquor at the outlet to the still to less than .005% of NH_3 , beyond which the makers



FIG. 17.—SHOWING METHOD OF REMOVING BUBBLING HOODS IN HOLMES STILL.

find that it does not pay to recover this small percentage of ammonia, as the additional lime and steam required, and the additional labour, are not compensated for by the small additional percentage of ammonia recovered.

Fig. 15 shows a small circular still, the overflows being seen with the doors removed, and Fig. 16 a large square still, showing the cleaning-out doors on the side of the still, and the overflows.

In place of the ordinary form of baffle box on the ammonia pipe leaving the still, which frequently is a cause of blockage and interference with the balance of the apparatus, a special hood is supplied having the form of a breeches pipe. This device acts more or less as an expansion chamber, and prevents any priming of the still without baffling the free exit of the ammonia gases, and there are no drain pipes necessary as in the case of a baffle box.

The best results in working these stills, as regards percentage of ammonia recovered and the quality of salt, have been obtained in plants fitted with the makers' bottom discharge saturator (Fig. 18), due to the fact that more intimate contact can be obtained between the ammonia gases leaving the cracker pipe and the acid in circular vessels of this description than in rectangular ones. The salt is also of a finer quality, and dries very readily. The bottom discharge type of saturator (Figs. 18 and 22) is designed with a cast iron external casing, the casing being perforated in order to indicate readily any leakage occurring from

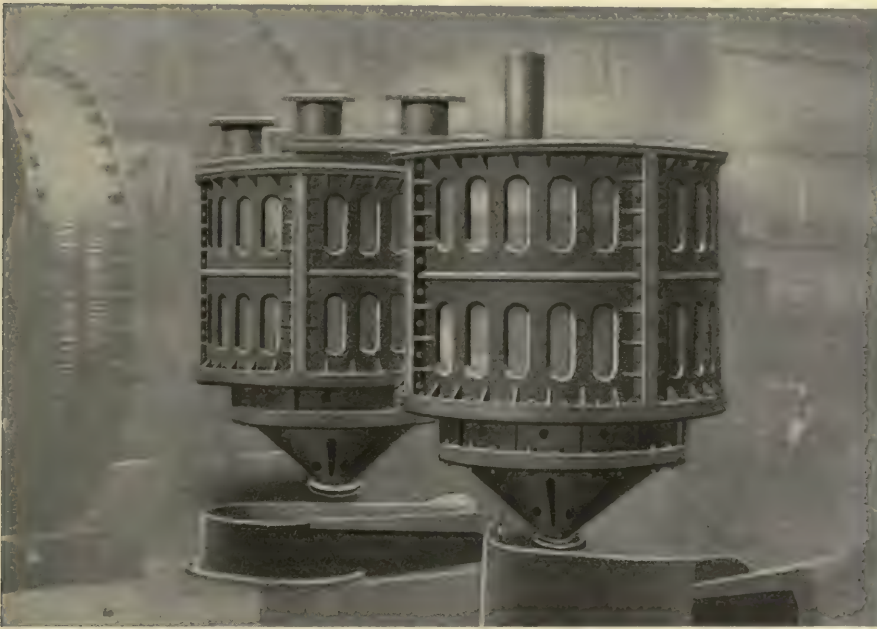


FIG. 18.—PAIR OF BOTTOM DISCHARGE SATURATORS BY W. C. HOLMES & CO., LTD.

the lead lining, and is made sufficiently strong to be independent of the strength of the lead lining, so that the latter receives adequate support, even when considerably worn. These saturators are made to work with ejectors and also of the fishing type, and very satisfactory results have been obtained, although in the case of the ejector it is found that there is a considerable amount of additional mother liquor formed which requires to be worked up.

Fig. 20 shows a sulphate installation in three sections, each distilling 100 tons of liquor per 24 hours. The apparatus is specially designed with a view of working up the liquors containing a small percentage of NH_3 , but can be modified in arrangement and design to work up

the liquors of any higher strength either for the manufacture of sulphate of ammonia, crude concentrated liquor, or pure liquor ammonia.

Fig. 22 is a view of a pair of bottom discharge saturators with centrifugal drier, producing 6 tons of salt per day.

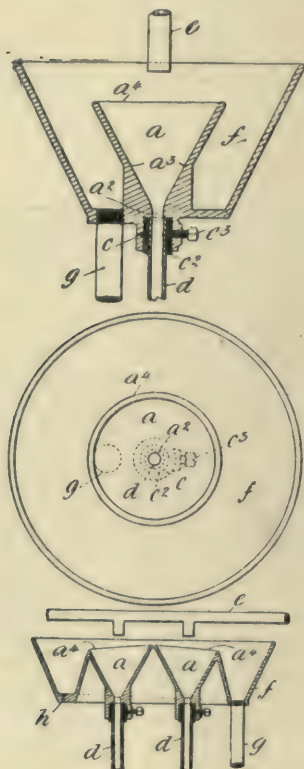


FIG. 19 — SHEWRING'S PATENT
CONTINUOUS SIGHT-FEED LIMING
APPARATUS.

Figs. 19 and 21 show the continuous sight-feed arrangement for the liming of sulphate stills (British Patents Nos. 11,777 and 21,301 of 1914) of Mr. Frederick Shewring, gas engineer. The patented apparatus is made by Messrs. W. C. Holmes and Co., Ltd.

Fig. 19 shows the details of the liquid receiving and regulating tanks. The receiver is an upright vessel of cone shape, from the bottom of which proceeds an outlet, and the second receiver is an upright vessel with cone sides surrounding the first receiver. E is the feed pipe and C the overflow. The overflow liquid passes over the rim of the inner receiver into the outer compartment, and passes away down the overflow pipe.

The outlet passage from the extreme bottom of the first receiver is formed without shoulder, rim, or any part which would retard the free flow of the liquid and tend to wipe out any solid matter in suspension. The size is determined to always maintain an overflowing condition into the second or outer receiver.

The patent mechanical agitator shown in Fig. 21 consists of substantially horizontal members of open-work construction, to which are secured a number of metal springs or wire coils. The horizontal members are preferably trough cross section, and they have a lifting action on the liquid when in operation. In the operation of the agitator the springs or coils are most effective in disturbing any settled solids and keeping them in suspension, also in preventing any undue strain on the mechanical devices by reason of the springs or coils compressing, and enabling the full depth of the tank to be reached with always a smooth and soft working of the agitator. The agitator operates with simultaneous upward, downward, and sideway motion. The following is a brief general description.

Reference to Fig. 21 will show the process fairly clearly. Lump lime is placed in the mixing tank, and spent liquor from the sulphate

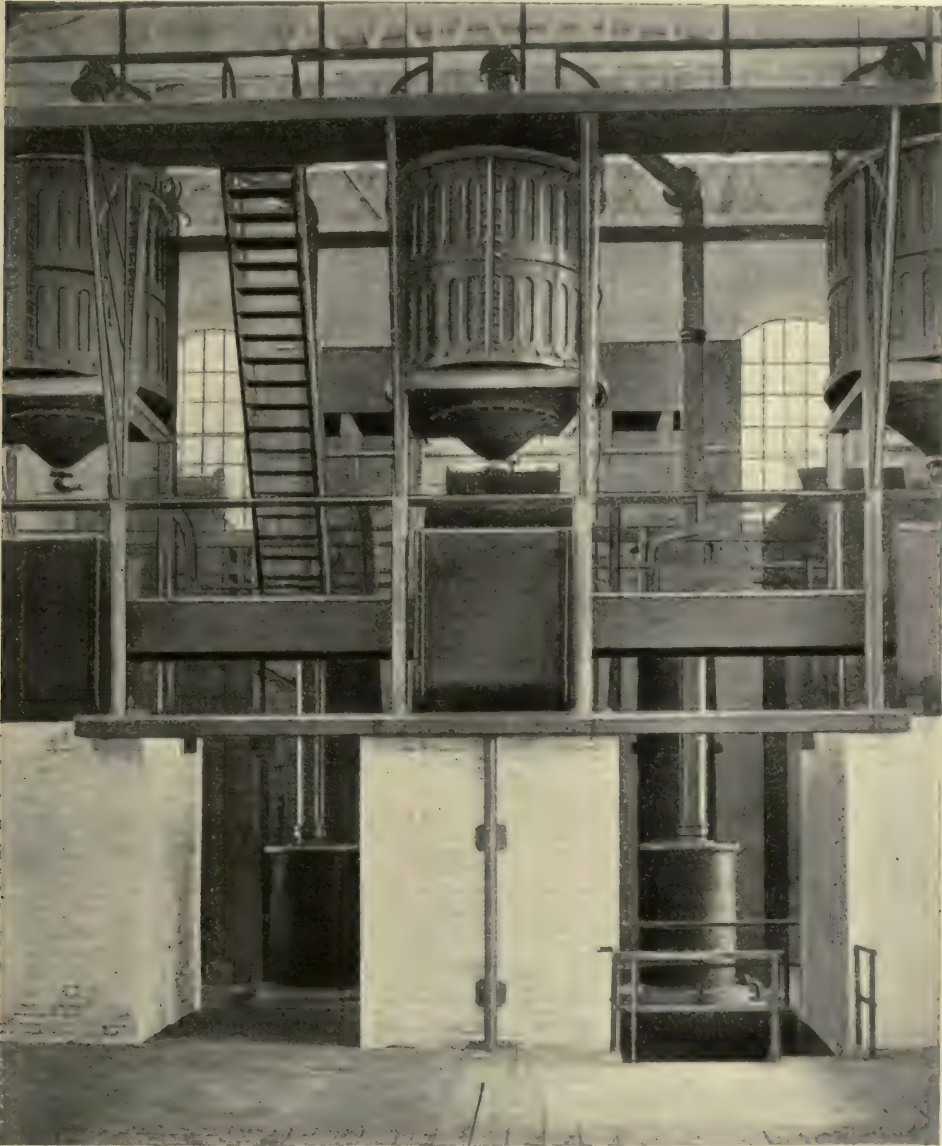


FIG. 20.—SULPHATE OF AMMONIA INSTALLATION BY W. C. HOLMES & CO., LTD.

plant is utilised for slaking purposes. When the lime is thoroughly slaked, the water spray is turned on, and washes the milk-of-lime through the strainer. The milk-of-lime is then run into the lime tank,

and is kept continuously agitated by the patent mechanical agitator. The liquid is lifted into an overhead regulating tank, which is so constructed that the soluble solids do not settle out of the milk-of-lime before passing to the still. The quantity of solution entering the still is under control, irrespective of the delivery to the overhead tank, and the attendant in charge of the sulphate plant can tell at a glance if the stills are being limed. The surplus milk-of-lime returns down the overflow pipe to the lime tank.

With this liming system it is possible to supply regulated feeds to one or more stills without having to duplicate the plant. The only modification entailed would be in the construction of the overhead receiving and regulating tank.

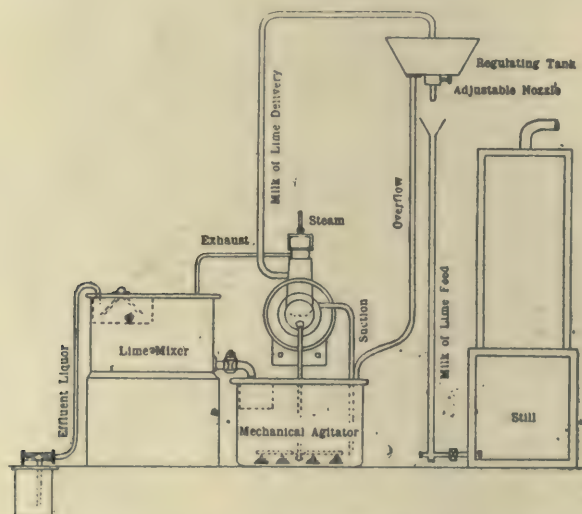


FIG. 21.—SHEWRING'S CONTINUOUS SIGHT-FEED APPARATUS.

The Scott Ammonia Still.—In the Scott still (British Patent 3987 of 1900), the ammonia liquor is heated under vacuum by means of open steam, after lime or other chemicals have been added if required. The vacuum causes the ammonia to come off more readily than it does under atmospheric pressure, and with the use of less steam. In the absorption of the ammonia in the saturator considerable heat is generated by the reaction, some of the water from the sulphuric acid solution is evaporated, and this steam, together with the steam which has come over with the ammonia, is conveyed into another ammonia still under a somewhat higher vacuum than the first, where more ammonia and steam are formed, to be treated as above described. This operation may be repeated several times. The incoming liquors are heated by the outgoing



FIG. 22.—PAIR OF BOTTOM DISCHARGE SATURATORS, WITH CENTRIFUGAL DRIER, BY MESSRS. W. C. HOLMES & CO., LTD., PRODUCING 6 TONS OF SULPHATE PER DAY.

liquors, so that a comparatively small quantity of steam is required to bring them to the boiling point.

Fig. 23 is a sectional elevation of suitable plant, reproduced from the Patent drawing.

The ammoniacal liquor is pumped into the heater *a* at the inlet *b*, and is forced through the heater *a*, which constitutes the bottom of the first vessel *c*. On arriving at the upper part of the heater, the liquor passes up by the external pipe *d* through a spring relief valve *e* into the top of the vessel *c*, which is arranged as an ammonia still, and which is under the influence of a vacuum. This liquor then passes down over and through the plates *c*¹ in the vessel *c*, running from one to the other of the plates, and meeting a current of steam, which causes the free ammonia to be driven off, and which is supplied through the inlet *f* at the bottom of the vessel *c*, into a coil not shown in the drawing. As there is a partial vacuum in the still *c* the steam may be exhaust or waste steam. The spring relief valve *e* is fitted in the pipe *d* to prevent the formation of ammonia vapour in the pipe from the effect of the vacuum. If vapour were formed it would interfere materially with the circulation of the liquor through the pipe. When the liquor contains carbonate and sulphate of ammonia, lime or soda is injected through the inlet pipe *g*. This lime or soda mixes with the partially freed liquor, and causes the fixed ammonia to be given off. The liquor continues its passage down the still *c* until it arrives at the bottom section. In its passage to this bottom section of the still the liquor has been boiled by the steam rising from the lower layers, and on arriving at the bottom is free from ammonia. It then flows through the bottom plate of the still into the heater *a*, the tubes of which it travels steadily, meeting the cold liquor which is being pumped in around the tubes through the inlet *b* as above described. By this means the boiling liquor is gradually cooled and the cold liquor is gradually heated. Further, the waste heat from the liquor is saved, the incoming cold liquor leaving the heater almost at the boiling point, and the outgoing hot liquor going away almost cold, so that comparatively very little steam of any kind is necessary. The natural tendency of heating and cooling liquors is observed, because the hot liquor as it grows colder is descending, and the cold liquor as it grows hotter is ascending, thus avoiding the formation of counter currents, which would be created if the heating and cooling liquors did not pursue these paths. *h* is an opening in the still *c* for receiving a thermometer.

The mixed ammonia vapour and steam generated in the still *c* pass over from the top of the still, through the pipe *i*, into the smaller vessel *j*, which must be of a material suitable for resisting the action of

sulphuric acid, such as copper, lead lined iron, etc. This vessel *j* contains sulphuric acid and sulphate of ammonia in solution. As the ammonia comes over, sulphuric acid is run in through the sulphuric acid inlet *k*, thus keeping the ammonia constantly neutralized, and preferably, generally, on the acid side. Sulphate of ammonia is immediately formed, and as the solution is saturated, solid sulphate of ammonia is thrown to the bottom of the vessel, and is drawn off from the influence of the vacuum, either continuously or from time to time by the discharging apparatus *l*, which may be of any suitable design. The vessel *j*, termed a "combination vessel," is under the influence of a slightly

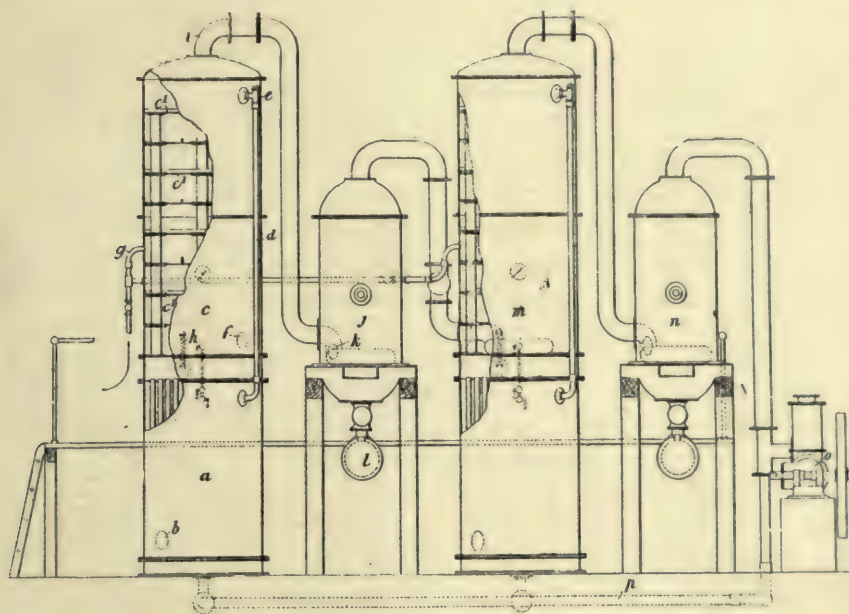


FIG. 23.—SCOTT AMMONIA STILL.

higher vacuum than is the first vessel, the difference of degree being that required to draw the steam and ammonia through the smaller layer of sulphuric acid and sulphate of ammonia in the bottom of the vessel. In ordinary work this amounts as a rule to a difference of about two inches of mercury.

Although the ammonia is arrested in this vessel, the steam passes on from the solution, which is always boiling. In addition to this steam, more is produced by the very large quantity of heat generated by the combination of the sulphuric acid with the ammonia. The extra water required for this steam is provided by mixing the sulphuric acid used with a sufficient quantity of water before allowing it to enter the appa-

ratus. The steam which is given off from this vessel will thus be seen to consist, not only of all the steam which comes from the first vessel, but also of a large quantity which is formed by the chemical combination. The whole of the steam passes on to the third vessel *m*, which works in exactly the same way as the first vessel or still *c*, except that it is under the influence of a higher vacuum, in order to obtain the necessary suction to cause the steam to pass through the layers of

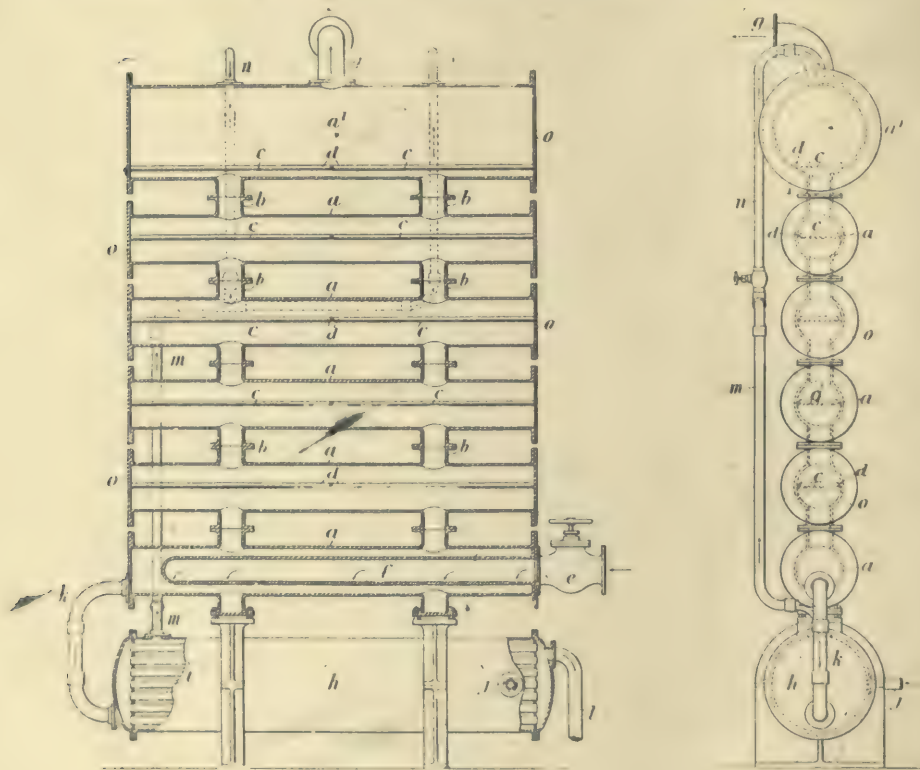


FIG. 24.—SCOTT AMMONIA STILL.

ammonia liquid under treatment; the steam from this third vessel *m* then passes on to the fourth vessel *n*, which works in the same way as the second vessel *j*, except that it also is under a higher vacuum than the second vessel. From the fourth vessel the steam produced is drawn over to the vacuum pump *o*, where a large proportion of the condensation may be effected by means of the cooled liquor drawn away through the pipe *p* from the bottoms of the vessels *c* and *m*.

Another form of apparatus (Fig. 24) is described in Scott's Patent 11,082 of 1901, in which the still is constructed of what may be described as

a series of pipes. Each of these pipes has one or more branches at the top and the same number of branches at the bottom, and inside each pipe is a perforated plate formed as a tray. The liquor to be treated is heated by the heater through which the hot spent liquor is passed, and is then delivered into the topmost pipe, passing into the tray; it then passes from the top of the pipe to the bottom through the perforations in the tray, meeting the steam and vapour which is rising from the pipe below as it does so. The liquor then passes through the branches at the bottom of the pipe into the next pipe, where it is delivered into another tray arranged in the same way. Here it meets the steam coming from the pipe below, and then descends into the third pipe of the still, and so on until it reaches the bottom. In the bottom pipe it is finally heated so as to remove the last portions of vapour, and passes out through the heater, where it heats the cold incoming liquor.

The left-hand drawing in Fig. 24 is a sectional side elevation of such apparatus, and the right-hand drawing is an end elevation. *a, a* are the pipes which form the body of the still, and *b, b* are the branches which are formed upon the top and bottom of the pipes *a*, the branches being flanged in order that they may be connected together so as to form the series of pipes into a complete still. The uppermost pipe *a* is connected by these flanges to a drum *a*¹, which forms a separating chamber for the mixed steam and vapour which rises through the still. *c, c* are the perforated plates provided inside each pipe and the drum so as to form trays in it. In practice each tray is made with edges *d, d*, and in two sections, so that each perforated partition is practically in the form of two trays, causing the liquor descending on each side of the still to remain within the limits of the two sets of trays on the right and left-hand side respectively, by which means the descending liquor is divided into two streams, a right and a left-hand stream. *e* is the steam inlet valve and *f* is the perforated pipe arranged inside the lowermost of the still pipes *a*, and through which the steam escapes into the still, rising up in the manner described above. *g* is the outlet through which the vapour distilled passes away from the still. *h* is a heater which is of ordinary construction, comprising a cylindrical casing having perforated end plates, between which are secured a number of tubes *i, i*. The liquor to be heated enters the cylindrical drum *h* through the inlet *j*, and is heated by the spent liquor which passes from the lowermost pipe *a* of the still through the pipe *k*, and through the pipes *i, i*, finally escaping through the outlet pipe *l*; the heated liquor is conveyed up through the pipe *m*, which is divided into two branches *n, n* entering the top of the separating chamber *a*¹ as shown.

With this arrangement the liquor from the heater *h* is introduced

into the separating chamber a^1 so as to fall upon the two perforated trays c, c provided therein; after passing through these trays it flows down the branches b, b , on to the trays d in the topmost pipe a . In this manner the liquor gradually passes from pipe to pipe until it finally, in spent condition, flows into the heater h through the pipe k to subject the cold incoming liquor to the preliminary heating. The steam and the vapour which have been distilled from the liquor under treatment, are separated in the separator a^1 , and the vapour escapes through the outlet pipe g to the saturator or condenser.

It will thus be seen that the still is built up in several equal sections which may be added to or reduced as may be desired. As there is no connection between the ends of the pipes, these pipes are closed with covers o, o , which can be removed when it is required to clean the still. When the end covers o, o are removed the internal trays d, d can be easily withdrawn, brushed, and replaced with facility.

The "Direct" Process of Ammonia Recovery.—During recent years a great deal of attention has been devoted by gas engineers to what is termed the "direct" process of ammonia recovery, which has been extensively adopted in the working of by-product coke-oven plants. The "direct" process consists, briefly, in the abandonment of the earlier practice of the recovery of ammonia by the absorptive power of water. The gases from the ovens or retorts pass, with or without cooling, direct to the sulphate house, where they are drawn by means of an exhauster through tar arresters, and then forced through sulphuric acid contained in a saturator similar to the ordinary type, the whole volume of the gas being thus treated. The advantages of this system of recovery were stated as follows by the Chief Inspector of Alkali Works in the Forty-seventh Annual Report:—"Operating in this way, the only liquor which has to be considered is such as may condense in the connections leading from the ovens to the tar arresters. It is of limited quantity, and can be dealt with so that no effluent need leave the work. Where the recovery of benzol is also aimed at, more thorough cooling of the raw gas is necessary, and a larger production of condensed liquor results, owing to the vapour of water present, but even then the quantity is comparatively small, and can be dealt with. In this way of working any noxious gases associated with the production of sulphate of ammonia are retained in the fuel gases, and pass along with these to be consumed in the ordinary operations of the factory."

Dealing further in the same Report with the application of the direct process to gas works practice, the Chief Inspector continues:—

"A system of ammonia recovery, similar in some respects to that just referred to in connection with coke-oven practice, was introduced during the year under review into gasworks. In both the whole volume of gas produced is passed through acid contained in a saturating vessel, and in both the discharge of an effluent liquor is avoided, but the conditions being different in the two classes of work the recovery plant differs in detail. In this system of working any sulphuretted hydrogen present proceeds along with the crude coal gas to be dealt with in the purifying plant before the illuminating gas is distributed to the consumers. A further advantage incident to this method of working which has a claim on the attention of all makers of sulphate from ammoniacal liquor is the absence of the necessity for storage and transference of such liquor. This advantage is twofold:—In the first place a possible source of much offence is avoided. In the past justifiable complaints have been made of objectionable effluvia arising from ammoniacal liquor stored in tanks. In the second place, opportunity for a tangible loss of ammonia is avoided. In the case of underground tanks this loss may arise and continue without being readily recognised. It is certain to arise, through the diffusion of the ever volatile ammonia, in the case of ammoniacal liquor tanks which have a ventilating communication with the atmosphere. Loss in this way will vary with the degree of exposure to the air, the strength of the liquor, the duration of storage, and from other influences."

This advantage of the direct as against the indirect or distillation method of recovery was further emphasised by the Chief Inspector in a communication on the subject to the *Journal of Gas Lighting* of November 23rd, 1915. In this communication the Chief Inspector gave details of experiments carried out at five works on the storage of ammoniacal liquor. The results showed that the loss of ammonia amounted to as much as 43.2% after eighty days, but that this loss could be almost completely avoided by covering the liquor with a coating of one-tenth of an inch of mineral oil. The Chief Inspector adds:—"The importance of this matter has been forcibly brought home to me by more than one instance coming within my knowledge where distillation of gas liquor kept in prolonged storage during summer months resulted in no separation of crystals of sulphate of ammonia in the saturator in the ordinary course of working. This seemed to be due to the very small proportion of ammonia remaining in the liquor when distilled. This possible source of loss should not be overlooked when comparing the direct with the ordinary method of sulphate of ammonia manufacture." The details of the experiments upon which these observations by the Chief Inspector are based, will be found in Appendix A,

The Wilton Direct Recovery Plant—One of the first gas works plants to be installed in this country for the operation of the direct recovery process was at Wareham. The plant is on the Wilton system, and was described by Mr. Philip G. Moon, F.C.S., in a paper read before the Southern District Association of Gas Engineers and Managers on November 10th, 1910. The plant deals with a make of gas amounting to about 5 million cubic feet per annum. The questions of capital expenditure and labour charges involved by the installation of sulphate of ammonia plant of the distillation type are serious matters in small works, and the plant in question is low in first cost, and does not entail any considerable extra labour.

The Wareham plant is shown in Fig. 25. The coal gas, after passing the condensers and a small purifier filled with heather, which acts as a somewhat crude tar extractor, enters the lead saturator A, and bubbles through the weak sulphuric acid.

It has been proved by careful experiments that there is no apparent loss of illuminating power due to the absorption of hydrocarbons from the gas, so long as the strength of the acid is kept below 4° to 5° Twaddel. In starting the acid washer, therefore, the acid is mixed to about 3° Tw. At this strength, the readiness with which it will absorb the ammonia renders it unnecessary to work with more than 1 inch to 2 inches of seal in the washer. Sulphate of ammonia is formed in solution, and the specific gravity of the washing liquor slowly rises. A simple calculation gives approximately the amount of sulphuric acid that should be added from day to day to neutralize the ammonia produced from the amount of coal carbonized per twenty-four hours. This amount is added to the saturator in half quantities each twelve hours. Roughly, about 23lbs. of acid of 142° Tw. would be required per ton of coal if all the fixed and the free ammonia had to be neutralized. Where the free ammonia only is dealt with, as at Wareham, about 13 to 16lbs. only is required. These amounts will, of course, vary according to the kind of coal carbonized and the heats used. Adding acid in this way, in the quantities necessary only for each day's make of ammonia, the specific gravity of the washing liquor increases regularly; but the amount of free acid in solution is never high enough to affect the illuminating power.

When the strength of the solution reaches 45° to 50° Tw., it is drawn off in small quantities each day, so as to keep the seal approximately constant, passed through the two small wooden, lead-lined filters BB (filled with sand on a layer of felt), and, when enough has accumulated, transferred to the evaporator C. This, at Wareham, where exhaust steam is available from the exhaust engine, is a wooden,

lead-lined vessel of the shape shown, with a flow-and-return lead pipe for the steam. Very little concentrating is needed, as at 45° to 50° Tw. the sulphate of ammonia solution is very near crystallizing point.

In works not using an exhauster, and therefore with no steam available, a cast-iron, lead-lined evaporator of convenient shape, sitting either on the waste-gas flue or on top of the beds, would be quite effective. Crystallization soon takes place, and the salt is put on the draining board D to drain, the small amount of mother liquor left being mixed with the next batch for concentrating. Very good coloured salt is made by this process with a percentage of 25.2% of ammonia.

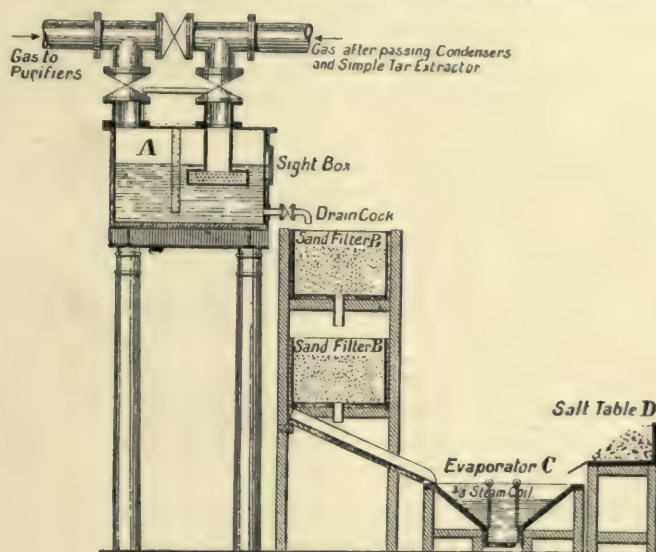


FIG 25.

In a small works such as that in question, the amount of virgin liquor from the hydraulic main is hardly sufficient to warrant the installing of a lime still to liberate the fixed ammonia. But the whole of this liquor, and the stronger liquor from the condensers, is pumped through the hydraulic main twice, so that as much free ammonia as possible is driven off into the gas. The virgin liquor run to waste at Wareham amounts to approximately 11 gallons per ton of coal, containing free ammonia equal to 1.1 lbs., and fixed ammonia equal to 4lbs. of sulphate. This is equivalent to a total waste of 5.1 lbs. of sulphate of ammonia per ton. Over a period of three months dealt with in the paper, 14 lbs. of sulphate of ammonia were made per ton of coal carbonized. In addition to this, gas entirely free from ammonia had been distributed, and the waste liquor to be disposed of had been considerably reduced in both strength and quantity. Mr. Moon remarks in his

paper that with a saturator of the kind described it is necessary with a process of this kind to keep the saturator liquor constantly on the acid side. If it were allowed to become alkaline, sulphuretted hydrogen and carbonic acid would be absorbed, and when the next acid was added, these gases would be given off in considerable quantities. The purifiers would be overtaxed by the burst of sulphuretted hydrogen, and the illuminating power of the gas would certainly suffer temporarily from the extra carbonic acid if oxide only were used for purification. Fortunately, it is quite easy to avoid this trouble.

The financial results of working the Wareham plant are thus summarised by Mr. Moon:—"As there is no fuel or labour bill to be met, practically the only charges against the sulphate sold are the cost of the acid used and the interest and depreciation on the plant itself. The capital outlay is, however, so small, and the wear and tear of plant so inappreciable, that these charges do not amount to very much per ton. I estimate that, selling the sulphate of ammonia produced at £11 per ton, there will be a net profit of £7 per ton sold."

The Wareham plant was mentioned by the Chief Inspector of Alkali Works in the Forty-Seventh Annual Report, already referred to. The Chief Inspector observed that hitherto no plant had been devised which was sufficiently simple and inexpensive for a small works to adopt. After describing the plant, and giving the particulars quoted above, he continued:—"It has been suggested that in smaller works a small stream of caustic soda or soda ash might be run into the hydraulic main to drive off the fixed ammonia, and experiments on these lines are being conducted at Wareham. Absorption of ammonia in weak sulphuric acid is not a new process, and it appears to have been tried at the Tottenham Gasworks in the forties by Mr. Alexander Croll. It was, however, discontinued, and it would appear that the sulphate of ammonia manufactured was of a bad colour; but at Wareham the difficulty of tar is overcome by the provision of means of filtration, and there is never any great deposition of tar in a weak solution of acid. A larger plant has been installed at Dunstable, and the method is being further extended by the erection of a plant at the works of the Woking District Gas Company. These larger plants are more satisfactory, inasmuch as they aim at collecting and utilising the whole of the ammonia driven off from the coal. Indeed, it is considered that the opportunities for leakage of ammonia are much less than with the usual arrangement of plant, seeing that storage tanks for liquor, in the ordinary sense, are dispensed with, the working up of the whole of the ammonia taking place continuously as it is produced. These larger plants are furnished with a direct-fired still, whereby the whole of the ammonia is recovered, so that a

better technical result is obtained, at the cost of an increased capital outlay and greater cost of working. Although this new method for sulphate of ammonia manufacture is suitable for larger works, yet it is probable that it will be appreciated by smaller works, isolated in position, but placed in the midst of agricultural districts where the local sale of sulphate of ammonia is advantageous. The Wareham method, with its low initial capital cost, and ease of operation without the necessity of any great amount of attention, will be a boon to those smaller gasworks where considerable difficulty is experienced in the disposal of gas liquor. It is, therefore, probable that the manufacture of sulphate of ammonia will now be carried on at many of those smaller gas undertakings where hitherto there has been little hope of making a profit by its introduction."

The Dunstable plant, as described by Mr. Moon, is shown in Fig. 26. At Dunstable, between 2,000 and 3,000 tons of coal are carbonized yearly, and the fixed ammonia is sufficiently significant to be recovered. The gas as it leaves the hydraulic main is passed through a specially constructed condenser-washer, so arranged that, as the liquor condenses, it flows in a reverse direction to the gas, and leaves the apparatus warm, and containing very little free ammonia.

This liquor is conveyed to a direct-fired distillation apparatus, where it is treated with alkali and the ammonia driven off to the acid washer through which the main stream of gas passes. The spent liquor, which averages about 10 gallons to the ton of coal carbonized, is conveyed to a shallow tank on the top of the retort-bench, and is evaporated to dryness. It will be seen that the quantity of effluent by this system is only about one-fourth of that which would be produced by the ordinary method of water washing and steam distillation. Thus at a small works carbonizing, say, 10 tons of coal per day, there would only be 100 gallons of effluent to dispose of, and this, if desired, could be easily evaporated in the ash pans.

Referring to Fig. 26, B shows the fractional condenser-washer, consisting of six bubbling trays, each working with a seal not exceeding $\frac{1}{2}$ -inch. The hot gases from the hydraulic main enter the washer at the bottom, and pass upwards through all the six trays. The two top trays may be used as naphthalene washers by filling them with a suitable solvent, such as oil tar, which also serves to remove the last traces of coal tar from the gas. The middle two trays are provided with cold water circulating pipes, which may be adjusted so that the exit gases are reduced to any desired temperature. The bottom two trays receive the condensed liquor from the trays above, and also the tar and liquor from the hydraulic main. The hot gas bubbling through the liquor in the

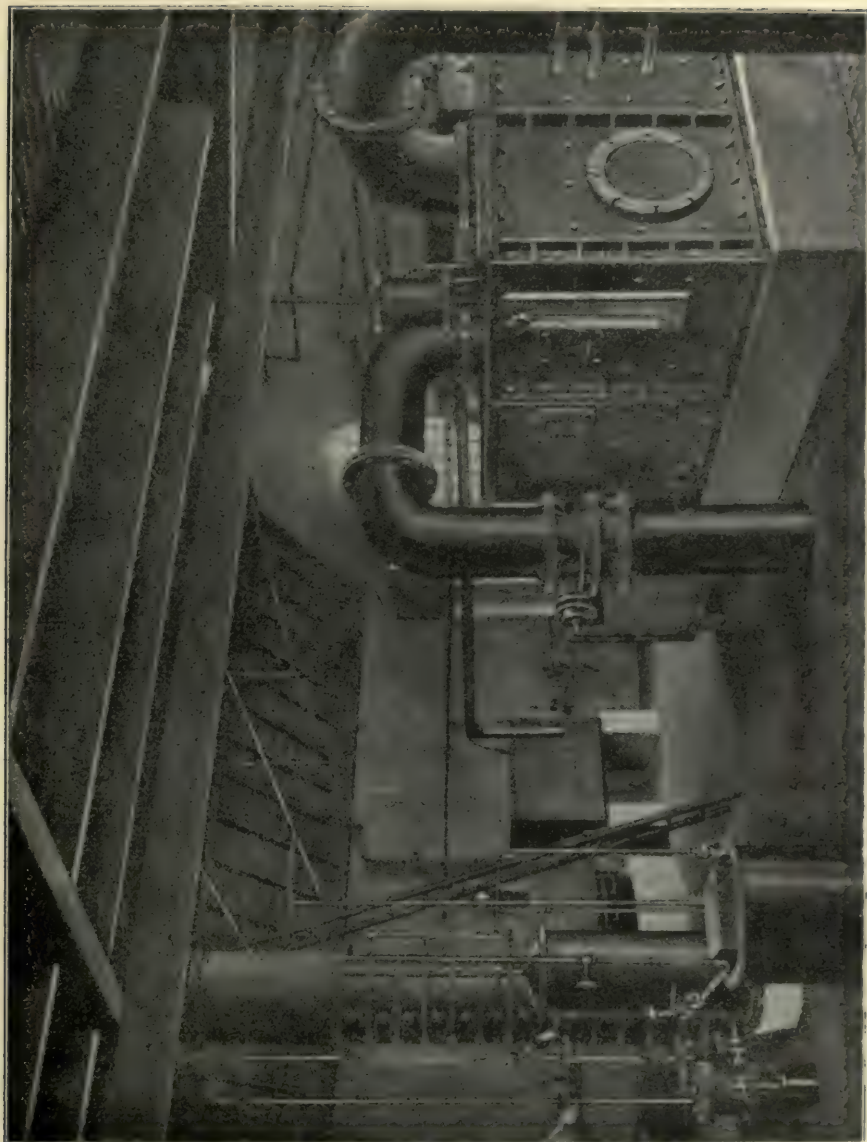


FIG. 27.—WILTON DIRECT RECOVERY PLANT AT MAIDENHEAD GAS WORKS.

Plate II. shows the complete Wilton plant for direct recovery as now in operation in a number of large gas works in this country, and Fig. 27 shows a typical installation at Maidenhead. The capacity of this latter plant is 60 to 100 million cubic feet per annum.

Figs. 28 and 29 give the details of the Wilton saturator for the direct recovery process as described in British Patent 3036 of 1909. The patentees observe that in the use of saturators of the ordinary construction the ammonia distributing pipes lie around the bottom of the saturating chamber inside the walls, and if such saturators are used for producing sulphate of ammonia direct from gas charged with ammonia

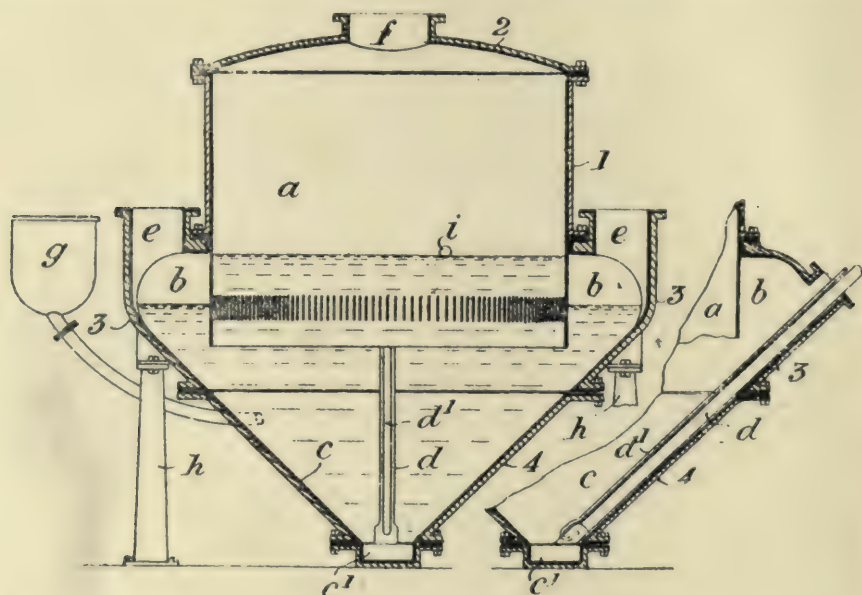


FIG. 28.

the pipes become choked with tar, and sometimes a mixture of tar and sulphate becomes piled up on top of the distributing pipes. It would not be a difficult matter to wash out the sulphate with water, but the tar is difficult to remove in this way.

In using saturators for making sulphate of ammonia from gas direct the saturators are necessarily of much larger dimensions than usual, to secure a large bubbling area, and such large saturators are expensive and inconvenient to make with a lead construction. The construction covered by this Patent provides a large bubbling area in a comparatively small saturator, whilst no corners exist in which the tar can become deposited with the crystals, and provision is also made for the ready removal of the tar.

The saturator is formed of an upper chamber freely open at its base, and surrounded or enclosed at or near its lower edge by a gas channel, both the gas channel and the upper chamber opening beneath, without obstructions, into a common settling tank, which is filled with the acid solution, in which the lower edge of the upper chamber is immersed. The arrangement is such that the gas in the gas channel

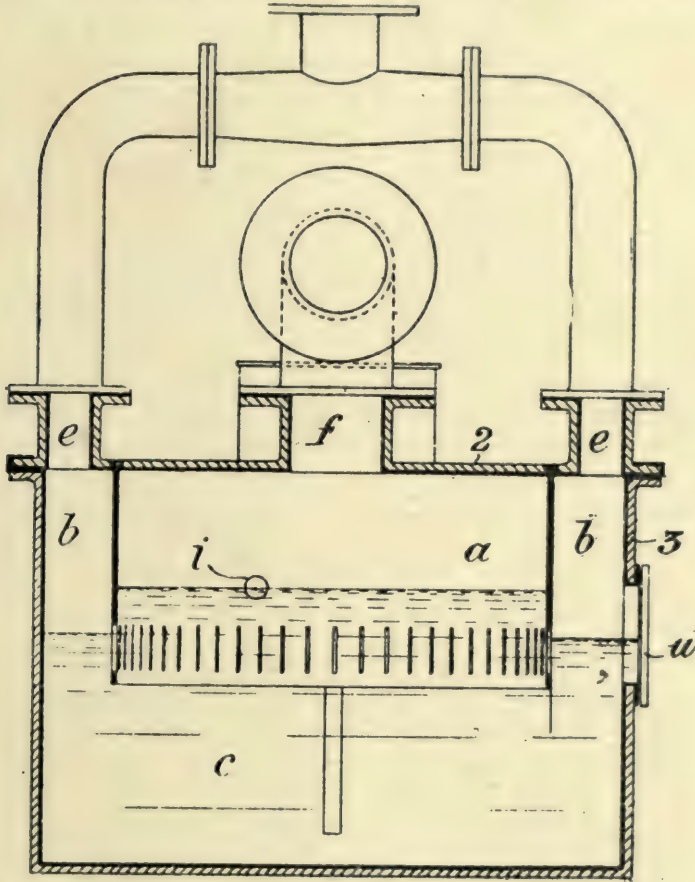


FIG 29.

bubbles under the edge of the upper chamber, which edge is formed with apertures or serrations for this purpose. By reason of the pressure of gas in the gas channel the liquid level in it is lower than the liquid level in the upper chamber. The tar which may be washed out of the gas will float on the surface of the saturator in the upper chamber, and can be drawn off by a pipe situated at the liquid level, more liquid being introduced for this purpose when required.

Fig. 28 is a sectional elevation of the saturator. The upper cham-

ber *a* is circular, and is surrounded by an annular gas channel *b* at its lower edge as described, this channel forming the upper edge of the tank *c* beneath, which tank may be of a more or less conical formation as shown, and formed with a recessed base *c*¹ in which the base of the usual sulphate of ammonia discharger or ejector is placed. The discharge pipe *d* and air or steam pipe *d*¹ of the discharger is led upward through the side of the chamber *c* in a suitable position as shown, or through the roof of the upper chamber *a*. Suitable gas inlets *e e* are provided in the upper part of the gas channel *b*, and a gas outlet *f* provided in the upper chamber *a*. The inlet for the supply of solution is arranged in the usual manner with an extension above the liquid level as indicated at *g*.

The whole apparatus may be built of iron lined with lead, the lead lining being carried into the flanged joints, that part of the upper chamber *a* which extends into the lower one to form the inner wall of the gas channel *b* being in the form of a circular apron made entirely of lead, copper, or other suitable alloy or substance. The apparatus may conveniently be built up of jointed sections, namely, (1) a cylindrical shell forming the walls of the upper chamber *a*; (2) a cover jointed to the shell; (3) the upper part of the lower conical tank *c* and gas channel *b* jointed by flanges at the upper edge to the cylindrical shell *a*, and (4) the base of the conical tank *c* suitably flanged and jointed to the upper part, the sides of the conical tank *c* being supported by suitable brackets or standards *h* from lugs on the upper part of the tank; *i* is a pipe for drawing off the tar.

If the saturator is to be employed for making a saturated solution which is to be discharged and made into crystals in another vessel, the tank may be formed with a flat base, the saturated solution being run off as required by a suitable pipe.

Fig. 29 is a sectional elevation showing one such construction, in which the saturator is made in two parts, and the cover of the upper chamber *a* is for convenience made level with the top of the gas channels, the tank *c* being formed with a flat base, and an inspection window such as *w* may be provided.

The further consideration of the direct recovery process must be undertaken in connection with by-product coke-oven practice, in which the adoption of the direct (or, in some instances, the semi-direct) method is now general. The design and construction of the by-product coke-oven itself is fully dealt with in the next chapter, but for convenience of arrangement the various systems adopted in connection with the different types of oven will now be described.

The Simon-Carves Direct Recovery Plant—The Simon-Carves

direct recovery plant is shown in Fig. 30. The gases from the ovens, collected in the hydraulic main A, are led through the foul gas main B into the extractor or cyclone C, shown in detail in Fig. 31. From this the gases pass through the dynamic extractor D (see also Fig. 32), the tars from both extractors being collected in the collecting tank E. The gases, which are then free from all traces of tar, are forced by the exhaustor F through the acid seal of the saturator G. After leaving the saturator, the gases, still hot, are returned to the ovens and burnt in the heating flues. During the whole process the temperature of the gas does not fall below the dew point, so that no condensation of the water vapours takes place. In cases where the cooling surface of the plant is too great, part of the gas main is covered with non-conducting material. In order to ensure continuous working, the final tar extractor and the saturator are duplicated. The pumps H circulate a small quantity of tar through the hydraulic and foul gas mains and through the extracting plant, to keep the pipes and apparatus clean.

The extraction of the tars by means of centrifugal force is a special feature of the

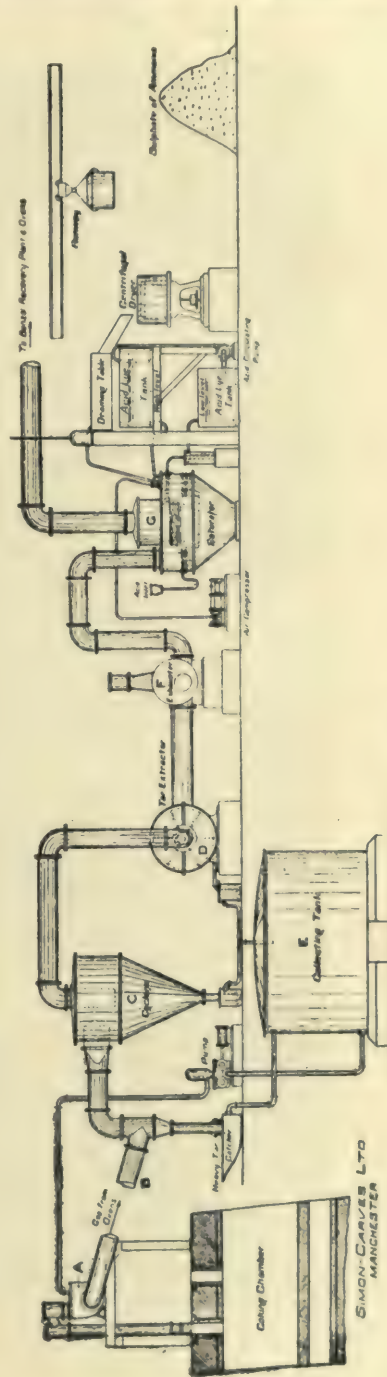


FIG. 30.

Simon-Carves process. The extraction is carried out in two stages; the removal of the heavier and denser tars being effected in a centri-



FIG. 31.—CYCLONE TAR EXTRACTOR, SIMON-CARVES DIRECT RECOVERY PROCESS.

fugal separator of the cyclone type, without moving parts (Fig. 31), and the lighter and more fluid tarry matters still contained in the gas

are then separated at a high velocity in a dynamic centrifugal separator. At the temperature at which the gas is treated in the tar extract-

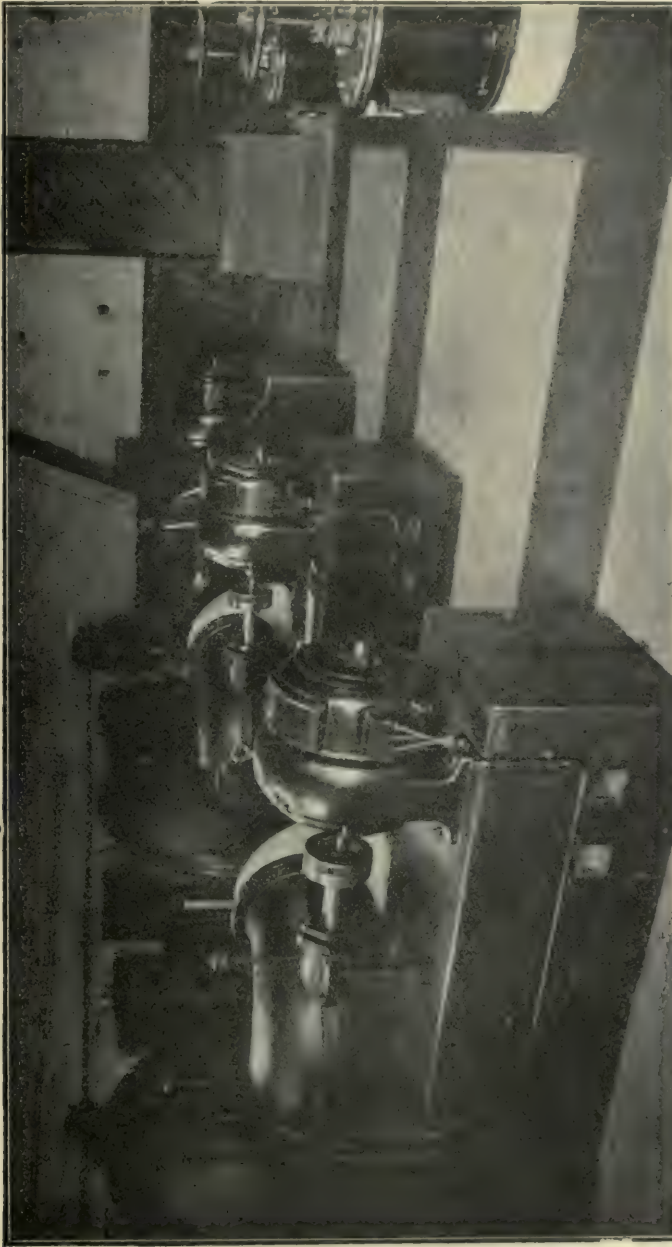


FIG. 32. DYNAMIC CENTRIFUGAL TAR EXTRACTORS AND MOTORS, SIMON-CARVES DIRECT RECOVERY PROCESS.

ing plant the tar vapours have condensed into very small drops, which are carried in suspension in the gas. The size of these drops varies

with the specific gravity of the tars, the heavier tar oils forming the larger drops. When a gyratory motion is imparted to the gas in a centri-

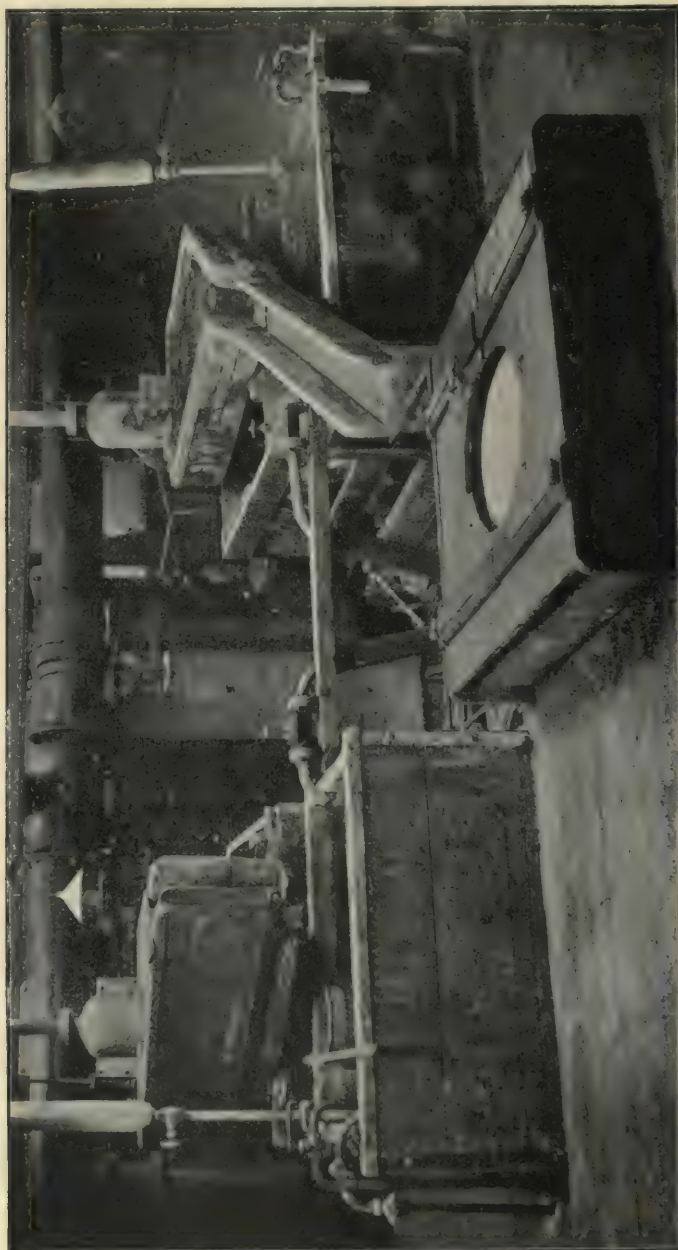


FIG. 33.—INTERIOR OF SULPHATE HOUSE, SIMON-CARVES DIRECT RECOVERY PROCESS.

fugal extracting apparatus, the resulting centrifugal action is much greater in the case of the tar drops than in that of the surrounding gas,

owing to their higher specific gravity. The tar drops are therefore thrown to the periphery of the centrifugal separator, where they coagulate, and can be drained away. It has been found that the drops above a certain size formed by the denser tars can be separated from the gas at a relatively low velocity, whereas the separation of the very fine drops formed by the lighter tars can only be effected by means of a very intense centrifugal action.

The cyclone tar separator is so designed that the action of the exhauster imparts a rotary motion, which is maintained without the aid of any moving parts until it leaves the apparatus. The gas enters the upper portion of the cyclone, which is cylindrical, through a tangential inlet at a velocity regulated by a valve of special construction. The conical shape of the lower portion of the cyclone counteracts the gradually diminishing velocity of the gas as it proceeds towards the bottom, so that in a well-proportioned apparatus the centrifugal action is fully maintained. The gas leaves the apparatus through a central outlet pipe which extends a certain distance below the top cover so as to prevent any interference with the cyclonic action. The tar which is separated from the gas by the centrifugal force developed collects at the bottom of the inverted cone, and flows out through a sealed drain pipe.

In the dynamic centrifugal separator a very rapid rotary motion is imparted to the gas by means of vanes of special construction revolving at a high speed, the peripheral speed being over 200 ft. per second. The fine drops of tar still contained in suspension in the gas after passing the cyclone are thrown on to the periphery of the separator, and being brought into violent contact with one another, combine into larger drops, which collect in the lower portion of the apparatus and are drained out.

As in the ordinary sulphate plants, the saturator is duplicated to ensure continuous working. The rest of the plant, *i.e.*, the draining tables, centrifugal drying machine, and sulphate store, is designed on the same lines as in sulphate plants working on the ordinary system. Fig. 33 is a view of the interior of the sulphate house, showing the general arrangement of the plant.

It is claimed for the Simon-Carves process that by its use it is possible to isolate the ammonium chloride contained in the gas at a very low cost. Where it is found advisable to recover chloride, a small quantity of liquor is allowed to condense in the dynamic separator, which washes the chloride out of the gas. On concentrating the solution thus obtained, chloride of ammonia is produced.

The "Otto" Direct Recovery Process.—The plant used in the operation of the "Otto" process for the direct recovery of ammonia and benzol is shown in Plate III. The process is fully described in a paper read before the Institution of Mining Engineers by Mr. Ernest Bury, from which the following extracts are taken.

Briefly, the process consists in extracting the tar from the hot moist gas above the dew-point, and then forcing the purified gas straight through sulphuric acid, where the ammonia combines with the acid and forms solid sulphate. On emerging from the acid bath, the hot gas returns to the ovens, or other points of consumption, still containing all its moisture in the form of vapour.

The raw gas is conducted from the oven-crown through a cast-iron ascension-pipe into the foul-gas and suction-mains, through which a small quantity of tar is continuously being circulated for the purpose of collecting and removing the pitchy matter deposited. These hydro-carbon bodies, on account of their possessing boiling points above 600° F. (320° C.), are mechanically carried forward in bulk by the hot raw gas, and partly precipitated in solid form in the mains, an action which would eventually obstruct the passage of the gas, but which is counteracted by the solvent properties of the continuous flow of tar. When the temperature has dropped to 212° F. (100° C.) or thereabouts by natural atmospheric cooling, the raw gas enters the tar-extractors. These consist of injector nozzles and a throat of certain form and dimensions, in connection with a tar-collecting tank and circulating pump. The raw gas is sucked through the throat by a stream of tar, which, on issuing from the spraying nozzle, assumes a conical shape, and penetrates the gas at all points in a shower of fine particles. This thorough mechanical treatment of the gas, together with the solvent action of the washing medium, results in the complete removal of the tar-fog, and the gas passes out from the extractors in a remarkably clean condition. The collecting tank into which the circulating tar gravitates is provided with an overflow to the storage-tanks, and with a connection to the centrifugal pump which re-circulates the washing medium. The temperature of the gas leaving the sprayers is usually between 150° and 170° F. (65° and 75° C.), the exact temperature depending upon the moisture-contents of the gas. As the object of the new process is to extract the tar-fog without precipitating the water-vapours of the gas, the sprayers are operated in such a manner as will ensure that the temperature of the gas does not fall below the dew-point, that is, the temperature at which the steam begins to condense out of the gas in liquid form. Besides this, the formation of liquor in the tar-sprayer soon becomes very detrimental to the cleaning action

of the tar-cone. After the foregoing treatment the clean gas is pressed forward to the exhausters, which are constructed to deal with the hot moist gas, and generally driven at a speed of more than 2,000 revolutions per minute, in order to force the gas through the acid seal in the saturator. The saturators (Fig. 35) are constructed of cast-iron framework, strongly lead-lined, with suitable dip-pipes for distributing the gas, and with an overflow into a scum-pot originally intended for the purpose of removing the skin or scum of tarry matters from off the surface of the mother-liquor, a process which nowadays, thanks to the

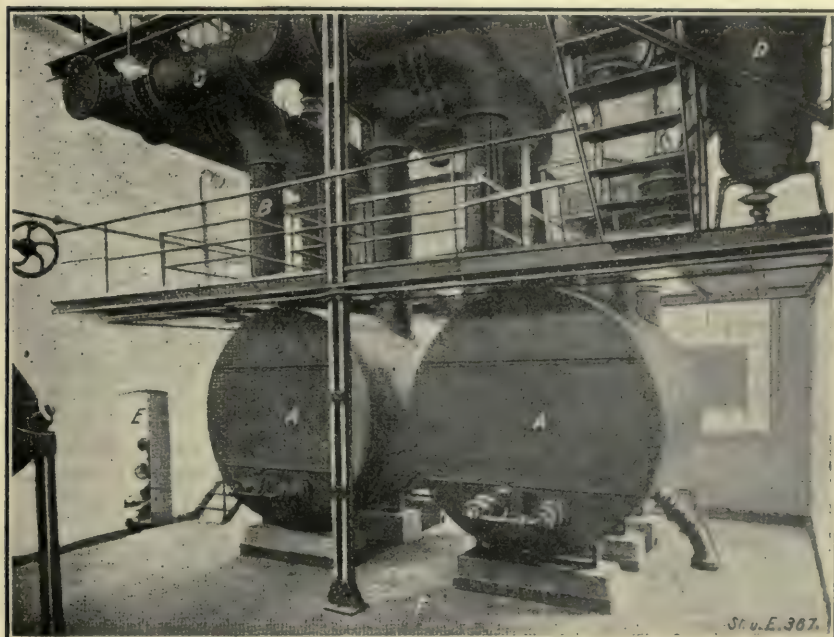


FIG. 34.—TAR EXTRACTORS, "OTTO" DIRECT RECOVERY PROCESS.

perfect washing action of the tar-sprayers, is only necessary at very long intervals. For instance, at the Carnock plant only two or three gallons of tarry liquid per month have been removed from the mother liquor. The heat generated in the saturator is more than sufficient both for preventing any condensation of steam in the bath, and for evaporating the water which is present in the sulphuric acid when fed into the saturator. The colour of the sulphate is a good grey, and in some cases almost white.

After emerging from the saturator, the hot gas, still containing all its moisture, is conducted back to the oven cellars, and distributed

in the manner common to the Otto ovens, namely, in cast-iron branch-pipes fitted with Bunsen burners underlying the vertical heating flues.

The following particulars of working results, from Messrs. Cochran and Company's New Brancepeth installation, are quoted by Mr. Bury:—The gas pressure in the hydraulic main averages 0.2 inch of water gauge, the suction on the main before the tar sprays is equal to 1 inch of water-gauge, and before the exhauster 4 inches of water-gauge. The pressure between the exhauster and the saturator is equal to 23 inches of water gauge, and after the saturators 4 inches of water gauge. The average temperatures are as under:—

| | °C. | | °C |
|----------------------|-----|---------------------|----|
| Hydraulic main ... | 225 | After exhauster ... | 66 |
| Before tar-spray ... | 86 | In saturator ... | 67 |
| After do. ... | 58 | After do. ... | 58 |

The General Manager, Mr. Peel, states that the ammonia in the return gas never exceeds 10 grammes per 100 cubic metres, and as there is no waste liquor, there is no loss in this way, as in the old method.

The sulphate of ammonia tests, on an average, as under:—

| | % |
|-----------------------------------|--------|
| Ammonia (NH ₃) | 25.20 |
| Moisture | 1 to 2 |
| Free acid | 0.44 |

With reference to the question of the avoidance of waste liquor and deteterious effluent, Mr. Bury observes that, under ordinary conditions the hot gas returns from the sulphate house to the ovens still containing all its moisture. If the return main is a long one, and not in any way insulated, a small quantity of condensings may be deposited by the gas before it reaches the cellar-burners. For instance, at the Alloa plant, under normal conditions of weather, hardly two tons are condensed in the whole length of the return gas-main (240 feet) during 24 hours. The latter is not covered in any way. Apart from the quantity being negligible, the nature of the condensings is much milder than that of ordinary waste liquor from stills. They contain no fixed lime salts, and approximate only a sixtieth of the total solids, a sixth of the total sulphur, and absorb only a quarter of the oxygen in a given time, as compared with the stills effluent, and they can be easily disposed of by being fed into the chimney-flue behind the boilers, or mixed with the water for coke-quenching purposes. The amount of water evaporated on the bench by a ton of red-hot coke equals approximately 10 cwts., and this is the most effective means of successfully disposing of all the condensings which are deposited by the gas after the satura-

tor, should the addition of benzol scrubbers enforce the necessity of cooling the gas to atmospheric temperature.

In a paper on "The Application of Coke-Oven Carbonizing and Otto Direct Ammonia Recovery to Gas-Works Requirements," read before the Institution of Gas Engineers (Liverpool Meeting, June, 1914), Mr. George Thomson Purves, A.M.Inst.C.E., of the Auchengeich Coke-Ovens, Chryston, gives some details of his experience in working the Otto direct recovery process. Referring to the spray tar extractor, Mr. Purves observes that this is a very simple but extremely efficient

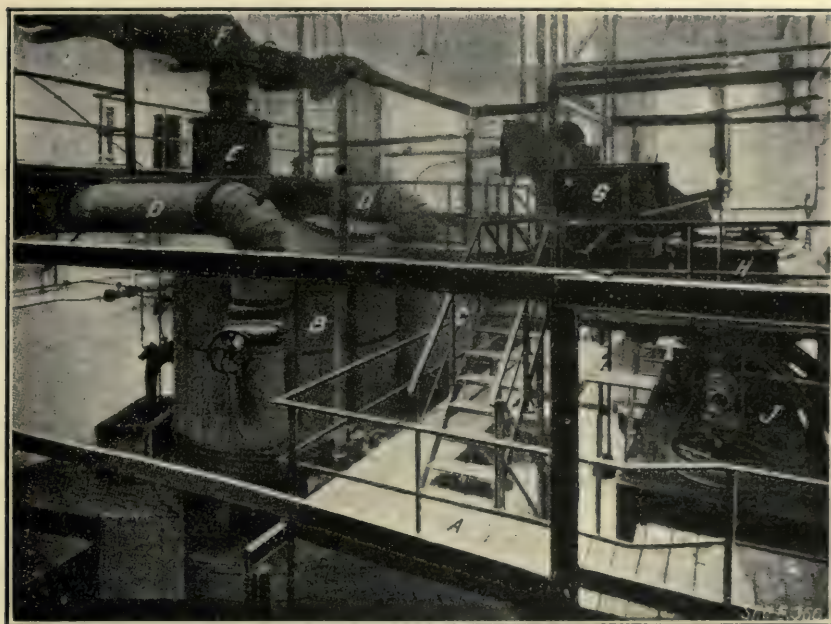


FIG. 35.—SULPHATE SATURATORS, "OTTO" DIRECT RECOVERY PROCESS.

piece of plant. The essential part in which the tar extraction takes place is a vertical cast-iron pipe, some 6 to 8 feet long, narrower at the top than at the bottom. The dimensions are carefully determined for the particular liquor jet which is to be used. It is the passage of the particles of liquor through the gas at very high velocity, and in such a manner that no gas can pass untreated, that effects the tar extraction. This apparatus is mounted on the collecting and settling tank, and the liquor circulation is performed by a "Weise and Monski" centrifugal pump direct-coupled to an electric motor. The liquor spray exactly fits the contracted pipe, and acts to some extent as an exhauster. The fine globules of liquor beat out the tar by bursting the tar envelopes just

as does the frictional contact in long condenser tubes. The only difference is that here the frictional surface moves, while in the other the gas moves. The tar and liquor collect in the tank underneath and separate, the liquor flowing back to the pump to be re-circulated, and the tar going to the deposit tank. The chloride of ammonia that is present in the gas is scrubbed out along with the tar, and dissolves in the liquor.

It will be evident that if the temperature of the gas and the ammoniacal liquor are just at the dew-point, neither deposition nor evaporation of liquor will take place. There is thus tar extraction without deposition of water. If the gas enters at a temperature above the dew-point, evaporation will take place, and so the liquor can be concentrated. In any case the gas will always leave the extraction plant at the dew-point. On the Continent tar instead of liquor is used in the extractor. Mr. Purves has tried creosote, tar, liquor, and tar mixed, and liquor alone, and found liquor the most satisfactory. When tar alone is used any ammonium chloride present in the gas is not so effectively scrubbed out as when liquor is used. The important points noticed here are that the extraction of the tar is purely frictional, and that the temperature of the liquor is too high for the free ammonia to be dissolved. Apart from the special case of chloride, a little fixed ammonia may slowly accumulate in the liquor. This can be pumped into the saturator from time to time, and the bulk of the circulating liquor made up with fresh water, or by condensation from the crude gas.

In gas practice various methods have been tried to remove the heavy tar from contact with the gas before completely cooling, with a view to preventing it absorbing light oil, and also with a view to reducing naphthalene trouble. Mr. Purves states that so far as he is aware, these attempts were without marked success. In the "Otto" method of tar extraction, there is a complete separation of the heavy tar from the gas while the temperature is still between 70° and 80° C. (158° and 176° F.), and with practically no extraction of light oils, as the following distillation test of the tar here deposited shows:

| | | | |
|--------------------------------------|---|---------|------------------|
| Up to 170° C. | - | 7.25 % | (water = 5.25 %) |
| 170° C. to 230° C. | - | 6.50 " | " " " |
| 230° C. to 270° C. | - | 13.50 " | " " " |
| 270° C. to 290° C. | - | 19.75 " | " " " |

The fraction up to 170° C. contained 5.25% of water, as the tar was not allowed to settle. The other 2% of this fraction consisted

almost entirely of naphthalene. The fact that this tar does not contain the light oils permits of its being kept hot with steam, until complete separation from liquor takes place.

The gas, free from tar-fog, but still carrying the light oils, now enters the saturator. The distance from the tar extractor to the saturator being only about 10 feet, any change in gas temperature is negligible. In passing through the saturator the gas is brought into intimate contact with the acid, being split up into fine streams by passing through a slotted curtain which hangs into the acid liquor. The curtain is circular in plan, and the gas enters on the outside, and leaves the saturator from the inside of the curtain. The depth of seal is adjustable. In the Auchengeich plant at the time of writing the paper it was 14 inches. The acid liquor in the saturator is kept in continuous circulation by means of centrifugal pumps, or else by hot acid injectors. The salt is ejected by means of air and steam.

The proportion of ammonium chloride entering the saturator is very small, and is not all decomposed, so that it is practically only the cooling due to radiation and conduction which must be corrected. After each make-up with acid, the temperature of the acid bath rises several degrees, and this excess over the average temperature is some time in being dissipated by the passing gases, etc. Continuous salting would seem a natural development in direct recovery, and with a well-designed and properly-lagged saturator and continuous salting, the heat generated by the chemical reactions would be nearly sufficient to account for all losses, and also evaporate the water added in the acid used. The ammonia passing the saturator and carried away in the gas amounts to about 1 oz. per ton of coal—better results are being obtained on more recent plants. The Auchengeich plant was the first direct recovery plant erected in this country. This small amount of ammonia passing being found sufficient to render the gas alkaline, no corrosion can take place from hydrochloric acid, and the amount of hydrochloric acid passing must be very small indeed. The tar-sprays in use at Auchengeich are the "Otto" old type, and are not so efficient as their latest type. A little chloride of ammonia gets into the saturator, but no corrosion has been observed, and none is feared. There is little doubt that, of the total chloride entering, only a small proportion is decomposed by the sulphuric acid.

The following analyses give a good idea of the composition of the acid liquor in its most acid state just after the make-up:—

| Acid liquor 60° Tw. at 84° C. | | | | | % |
|-------------------------------|-----|-----|-----|-----|-------|
| Sulphuric acid (free) | ... | ... | ... | ... | 11.06 |
| Sulphate of ammonia | ... | ... | ... | ... | 43.70 |
| Hydrochloric acid (free)... | ... | ... | ... | ... | .01 |
| Chloride of ammonia | ... | ... | ... | ... | 3.38 |
| Water | ... | ... | ... | ... | 38.75 |
| Other solids | ... | ... | ... | ... | 2.85 |

 99.75

If the gas enters the tar-extracting sprays considerably above the dew-point, tar-fog will be produced in the sprays, so lowering their scrubbing efficiency. A little tar will thus go forward to the saturator. From the samples of sulphate specially prepared under such conditions it will be seen how little this effects the colour even when the entering temperature is as high as 120° C.—that is, about 45° C. above the dew-point.

The Koppers Direct Recovery Plant.—Plate IV. shows a diagrammatic representation of the Koppers system of direct recovery. In this process, the hot gas from the ovens enters the preliminary cooler (A), which is of the multitubular type, and after passing through the tubes emerges at the bottom, and is led into the water coolers (B), where the temperature is reduced to about 25°C. The gas is then drawn by an exhaustor (C), by which it is delivered to the tar extractor (D). After having been freed from the tar the gas is returned to the preliminary cooler (A), where, in passing between the tubes, it becomes heated up, and at the same time exerts a cooling effect on the hot gas. The heated gas is then conducted along the main (F) to the saturator (E). The saturator is of the totally enclosed type, and the salt is continuously removed by means of an ejector, and delivered on to a collecting table, from whence it is run off, together with the accompanying mother liquor, into a centrifugal dryer (L) in the usual way.

The gas passes out of the saturator by the main (K), by which it is conducted back to the ovens and other points where required. In cases where benzol is required to be recovered from the gas, it is necessary for the gas to be cooled down after leaving the saturator, after which it is subjected to the usual scrubbing operation. It has been found from actual working experience at the plant in operation at the Mont Cenis Colliery, Westphalia, that the yield of benzol from the new system is greater than that obtainable by the ordinary system, which is due to the gas being practically free from moisture when it is passed through the benzol scrubbers.

The products of condensation which are extracted in the cooling and tar eliminating operations, are drawn off from the several apparatus, and are conveyed into a separating tank (H), where the tar and ammoniacal liquor separate according to their specific gravities. The tar flows into the storage tank (I), and the gas liquor into the storage tank (J). The liquor is pumped to the ammonia still (G), where the ammonia is driven off by means of steam and lime in the usual way. The vapours of distillation are conducted from the still, and are delivered into the main (F), where they mix with the heated gas, and the whole then passes into the saturator. It is claimed that the temperature of the acid in the saturator does not exceed $65^{\circ}\text{C}.$, and that in consequence of this low working temperature the wear and tear is considerably reduced.

In some notes on the working of the Koppers system (*Journal of Gas Lighting*, April 7th, 1914), G. Stanley Cooper says:—"In the low-temperature process it is contended that in order to produce the necessary conditions for the manufacture of a clean salt, there must be an absence of tar in the gas, and the temperature must be high enough in the saturator to prevent any dilution of the acid by water condensed from the gas, in order to allow of the production of solid sulphate direct, and not by evaporation. It is claimed that the Koppers process fulfils these conditions perfectly, and a detailed explanation of the process, together with references to the temperatures and pressures at particular points, will enable those interested to see exactly how it might be successfully applied to gas-works conditions. The temperature of the gas at the entrance to the coolers may be anything from $150^{\circ}\text{C}.$ to $250^{\circ}\text{C}.$, according to the length of the conducting main, etc. The gas at this point is usually under a suction of about 2 inches water-gauge. On passing through the coolers, the whole of the water vapour contained in the gas—above the saturation amount at the exit temperature—is deposited, together with the heavier portion of the tar. Hence the volume of gas, water-vapour, etc., is reduced at the exit of the coolers to about half what it was at the entrance to them; and the suction of 5 to 6 inches before the exhausters requires comparatively little power to maintain. The temperature at the outlet of the coolers is usually about $25^{\circ}\text{C}.$ The gas being drawn by the exhaustor is then forced through the tar extractor in order to remove all traces of tar fog, with which troublesome matter gas engineers are fully acquainted. Any form of tar-extractor can, of course, be employed. Generally either a Pelouze and Audouin or else a rotary extractor is used. The pressure before the tar separator is usually 30 to 40 inches. If the cooled gas is passed direct into the saturator, it is not an easy matter to produce solid sulphate of ammonia unless a very

strong acid bath is used. Even then it is often found necessary to supply heat from an outside source. Koppers introduces in his process a simple form of reheater. This consists of an iron box, inside which are coils of pipes through which exhaust steam passes. By this means the temperature of the gas is raised to about 60°C . The hot gas then passes into the saturator at a pressure of about 32 inches. The lye in the saturator usually contains from 5 to 10% of free acid. As pointed out above, by cooling the gas to 25°C . in the first instance the amount of water vapour in it is only sufficient to saturate it at that temperature. When, therefore, the gas is heated to 60°C ., it is capable of taking up much more water vapour; and this in fact actually happens. The gas passing through the lye in the saturator takes up moisture, concentrates the solution, and so helps the production of solid salt. After the saturator the temperature of the gas is about 55°C ., and the pressure about 12 inches. The gas is now totally free from tar and ammonia, but it contains much moisture. It is therefore necessary, before further purification takes place, to cool the gas once more to atmospheric temperature, and so remove the excess of moisture. The liquor condensed in the coolers will contain probably about 15 to 30% of the total ammonia, according to the class of coal being dealt with. Practically 80% of this ammonia will be in the form of fixed ammonia salts. This liquor is subjected to distillation with steam and lime in the usual way. It has to be remembered, however, that no water whatever is used for scrubbing purposes. Hence the amount of liquor is probably only about 30 to 40% of the quantity produced in the old method, and many of the objectionable constituents are absent. . . . The sulphate produced is of excellent quality, and quite white in colour. The following is a typical analysis: Ammonia, 25.3%; moisture, 0.2%; free acid, 0.3%; ammonium chloride, *nil*. The costs of operating such a system are not prohibitive. The labour required is not a great item. On a plant dealing with (say) 600 tons of coal per day, making approximately $7\frac{1}{2}$ million cubic feet of gas, the labour required would be only five men per shift. Assuming a yield of 1.4% of sulphate of ammonia, the production would be 8.4 tons per day, or approximately 3,000 tons per annum. For this would likely be required about 3,000 tons of sulphuric acid and (say) 350 tons of lime. An ample allowance for repairs and maintenance is $2\frac{1}{2}\%$, as owing to the low temperature at which the system is worked and the elimination by cooling of any corrosive elements, the wear and tear is reduced to a minimum. The whole process is simple and continuous, and requires little attention, all the operations being practically automatic. The power requirements of the plant are not great. Steam is necessary for the exhauster according to

the capacity of the plant, and the exhaust steam from this source is sufficient to serve for reheating the gas before the saturator, and for distilling the liquor produced."

The Semet-Solvay Direct Recovery Process.—In Semet-Solvay coke-oven plants it is the practice to conduct the gases leaving the oven through an offtake pipe, making a connection as short as possible with a hydraulic main, where they bubble through weak ammonia liquor. The object is to reduce to a minimum the deposition of tar and carbonaceous matter in "dry" mains, for long pipes mean labour in cleaning, loss of time between charges, and loss of recoverable by-products. A large circulation of liquor and thin tar keeps the hydraulic main clean.

In circumstances where the "direct" recovery process is essential the gases are collected in a tar main and no liquors are circulated. On leaving this main the gases pass through an apparatus for regulating temperatures, and are afterwards washed with tar in a series of bubbles (British Patent No. 7915 of 1910). The gases, now freed from tar, and still at a temperature above the point at which water is deposited, are then bubbled through sulphuric acid, where the ammonia is recovered in the form of sulphate, which is periodically ejected from the saturator, dried, and sent to the sulphate stores. In circumstances where effluent liquors cannot be got rid of the recovery of benzol must be sacrificed. If, on the other hand, means can be found for the disposal of such waste liquors, the ammonia-free gases may be cooled to get rid of the contained water, and washed with absorbing oil to recover the benzol. Semet Solvay plants on this system have been erected in Belgium, one at Quiévrain, and another at the works of the Société John Cockerill, at Seraing.

Troubles have been experienced with this form of the "direct" or "hot" process, in the formation, for example, of naphthalene on cooling, and the deposition of fixed ammonia salts in the tar and in the gas mains, and the latter has been of a somewhat serious character where the coals treated yield a high proportion of ammonia fixed as chloride. Although the ammonia thus fixed is recoverable when appropriate means are employed, the demand for chloride of ammonia is limited, and it is desirable to avoid producing it.

To avoid the difficulties inherent to the "direct" process the Mont Cenis process may be adopted. In this the gases are cooled to the point at which the tar and water (containing part of the ammonia, including all the fixed portion) are deposited, the ammonia liquors are distilled in the usual way with lime additions, and the vapours of dis-

tillation added to the gases. The gases and ammonia vapour are then passed through a direct saturator (in the Mont Cenis process this may be done without superheating the gases or vapours), and the sulphate of ammonia recovered in the usual way.

The gases are not scrubbed with water to recover the ammonia, as in the old practice. There is therefore a substantial reduction in the quantity of ammonia liquor to be distilled, and a corresponding diminution in steam and lime water additions and in waste liquors produced.

Figs. 36 and 37 show a new direct-recovery process which has been installed by the Coke-Oven Construction Co., Ltd., of Sheffield, in connection with the Semet-Solvay ovens in operation at the Beighton Colliery.

The gases from the ovens pass through a cooler in the usual

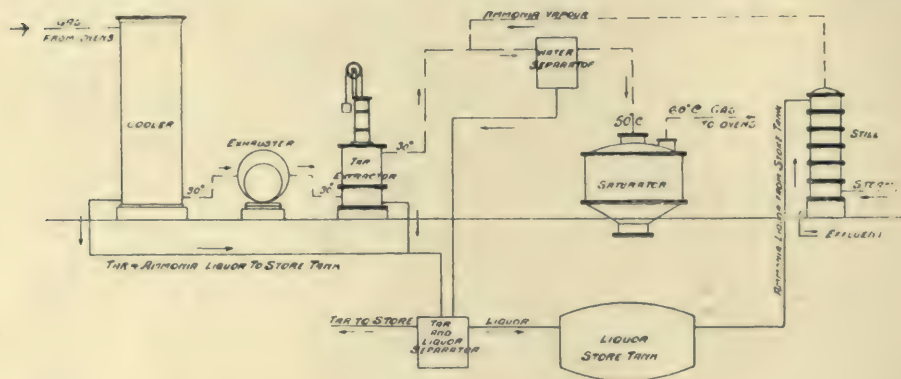


FIG. 36.—DIAGRAMATIC SKETCH SHOWING ARRANGEMENT OF DIRECT-RECOVERY PROCESS, WITHOUT BENZOL SCRUBBER, AT BEIGHTON COLLIERY.

manner, thence by way of the exhauster through the tar extractor, from which they are led direct to the saturator. The condensate from the cooler is led to the tar and liquor separator, from which the liquor overflows to the storage tank, and is thence pumped into a still, where it is treated with steam and lime, and from which still the ammonia vapour mixes with the main volume of gas on the feed side of the saturator. In all such systems the mixing of the cooled and partially purified gases with the ammonia vapour from the distillation column results in an interchange of heat, until the equilibrium temperature is reached, and the mixture is in a supersaturated state. The mixture of gases thus contains a certain amount of "water fog," corresponding to the degree of supersaturation, and, unless means are taken to avoid it, the gases would carry this "water fog" forward with them in their passage to the saturator, and the bath would be diluted to such an extent

that solid salt could not be obtained. A common practice, and one until recently adopted at Beighton, is to place a condenser or dryer in the path of the ammonia vapour, between the still and the point where it joins the gas main from the tar extractor.

In the system under notice the ammonia vapour is led direct from the still into the gas main, where it mixes with the volume of gases, the resultant mixture being passed through a water separator to remove the water fog, and thence directly into the saturator. The mixture is then in a purely saturated state, and there is a greater margin of temperature to allow for radiation loss than is the case with the super-saturated gases previously mentioned.

The removal of "water fog" was obviously the first consideration, but the arrangement accomplishes a very useful purpose in another

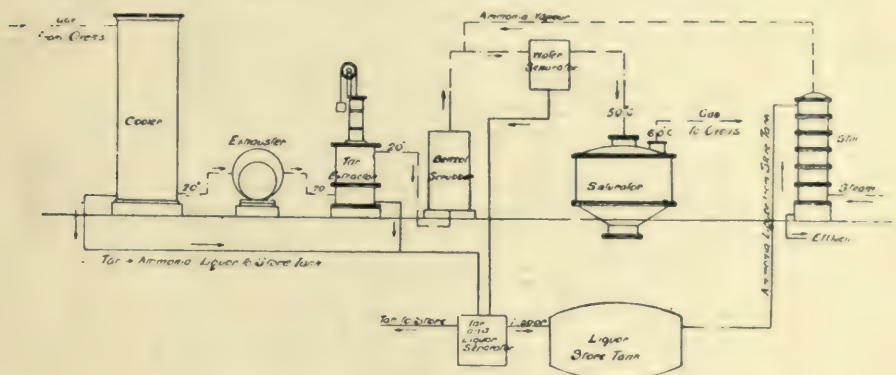


FIG. 37.—DIAGRAMATIC SKETCH SHOWING ARRANGEMENT OF DIRECT-RECOVERY PROCESS, WITH BENZOL SCRUBBER, AT BEIGHTON COLLIERY.

direction, as the separator serves to remove the last traces of tar, light oils, and other impurities, resulting in the production of a pure white salt.

The condensate from the separator drains back into the tar and liquor separating tank. The distillation column or still does not necessarily work continuously, but is usually laid off at the week-end, according to the make of liquor to be dealt with; nor is salt made continuously, the acidity of the bath being increased during alternate shifts to carry over until the next shift, when it is salted out. The production of salt is about 1,500 tons per annum.

As is well known, where benzol is recovered it is the practice in the case of the semi-direct process to re-cool the gases after they have passed through the saturator, and before they enter the scrubbers; but to avoid a second condensing plant and the increased water consump-

tion incidental thereto, the arrangement shown diagrammatically in Fig. 37 is installed at Beighton, and is giving excellent results. As shown, the benzol scrubber is interposed between the tar extractor and the ammonia saturator.

It is claimed that the new process has the advantage that the quantity of liquid treated in the distillation column per unit of gas made is considerably less than with the other processes; in addition, superheating of the gases is avoided, no cooling of the ammonia vapours is required, and owing to the gasses passing through the saturator being in a purely saturated state, the process is less dependent upon the ammonia content than is the case with gases passing through the saturator in a supersaturated state.

The Coppee Direct Recovery Process.—The Coppée direct recovery plant is shown in Plate V. In this plant the gas leaving the ovens is cooled down to about 90° F. in the ordinary way, and is passed through a Pelouze extractor for the purpose of extracting the tar. It then passes direct into an enclosed lead saturator. The small quantity of ammonia liquor which is produced by cooling the gases down to 90° F. is distilled in the still shown, and the resulting ammonia gas is passed into the gas-main immediately before the saturator, so that the whole of the ammonia goes into the saturator with the oven gas. The heat produced by the combination of the ammonia with the acid prevents any further condensation of vapour. The process is continuous, and only needs attention for adding fresh acid, and removing the sulphate. The sulphate is ejected from the saturator by steam or compressed air, drained on the draining-table, dried in a turbo dryer, and conveyed mechanically to the sulphate store. The waste gases, consisting largely of carbon dioxide and sulphuretted hydrogen, pass with the clean gas into the return gas main, and are led to the ovens.

The British Coke Ovens, Ltd., Direct Recovery Process.—The position of this process is indicated by dividing the direct recovery processes into two groups as follows:—Group "A": (1) Removal of tar vapours by scrubbing the hot gases with tar. (2) No cooling of the hot gases before passing into saturator, in order to avoid formation of ammoniacal liquor through condensation of water vapours. (3) Cooling gases prior to extraction of benzol with corresponding condensation of water vapours, discharged as waste liquor. Group "B": (1) Removal of tar vapours through cooling the gases. (2) Partial removal of ammonia (about 50%) through absorption by condensed water vapours, discharged as ammoniacal liquor. (3) Distilling the ammoniacal liquor by

means of steam, and returning the ammonia vapours into the main gas stream. (4) Reheating the gas stream prior to its passing through the

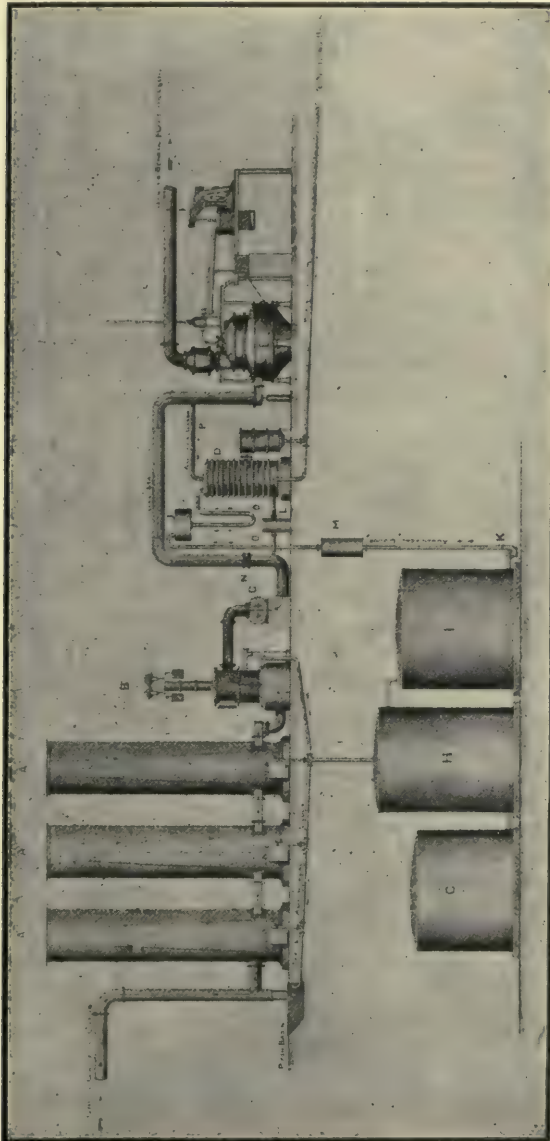


FIG. 38.—THE BRITISH COKE OVENS, LTD., DIRECT RECOVERY PROCESS.

saturation. The "B.C.O." process does not come into either of these groups, and it is claimed that it is an improvement on both. The principal difference between the "B.C.O." process and the Group B pro-

cess is in the method adopted for dealing with the ammoniacal liquor. This is explained as follows, reference being made to Fig. 38.

The crude gases coming from the coke ovens are cooled in the ordinary way in a series of water-cooled condensers, A, to a temperature below 70° F. before being drawn through the tar extractor, B. In this apparatus the gases are freed from the last traces of tar before being drawn towards the exhauster, C, and passed under pressure into the saturator, E. A throttle valve, N, is provided in the gas main immediately behind the exhauster, C, which is regulated in such a manner as to give the pressure required to ensure the supply of a suitable quantity of gas by way of re-heater L, and suitable pipe connections, O, into the bottom section of the ammonia still, D, where it is employed for the distillation of the ammoniacal liquor, steam therefore not being required. The temperature of the gas employed for distillation is preferably kept at about 212° F. The ammoniacal liquor coming from the condensers is separated from the tar in the settling tank, H, stored in tank I, whence it is taken, by means of the pump, K, and passed through a preheater, M. This apparatus is so regulated as to raise the temperature of the liquor on its way to the feed-tank, J, to about boiling point. The ammoniacal liquor flows from this tank into the upper tray of the still, D, and as it descends is stripped of the ammonia by the current of heated gas passing through the still in an upward direction, and the gas charged with ammonia is passed into the main gas stream through the pipe, P, and into the saturator, E. The ammonia is there recovered in the usual way, by absorption in dilute acid. The salt is ejected from the saturator and dried in the usual way.

It is claimed that a reduction of 15 to 20% on the amount of liquor entering the still is brought about in the "B.C.O." process. The plant is so arranged that the gas re-heater, L, and liquor pre-heater, M, are heated by means of flue gases (waste heat) coming from the ovens. No steam is, therefore, required in the "B.C.O." direct recovery process.

The Coal Distillation Company's Direct Recovery Process.—

Plate VI. shows the direct recovery plant of the Coal Distillation Company, of Middlesbrough. The plant consists of one or more water coolers A to cool down the gases coming from the coke ovens, a tar extractor B, an exhauster C, a saturator D, and ammonia still E, together with the necessary tanks.

The gas which comes from the coke ovens is cooled down to about 30° C. to remove the tar and part of its water vapour. It then passes through a Pelouze tar extractor, which removes the last traces of tar, and then goes through an exhauster. The compression in the

exhauster adds about 10° to the heat of the gas, and this rise in temperature is, together with the chemical heat developed in the saturator, sufficient to evaporate all excess moisture from the saturator, and to produce a solid sulphate of ammonia in the usual manner. The small amount of ammonia liquor produced through the condensation of water vapour in the gas in the coolers is from time to time distilled in the small ammonia still, and the ammonia vapour produced either added to the gas or sent to the saturator separately.

If part of the gas after the extraction of the by-products is used for illuminating purposes, it is advisable to let the ammonia vapour from the still, which is particularly rich in sulphur compounds, go into the saturator through a special seal pipe, which carries away the foul gases, and prevents the sulphur compounds from contaminating the cold gas.

Where benzol extraction is carried out, which is now the usual procedure, the gas, after leaving the saturator, passes into the benzol scrubbers, preferably after having been cooled down as much as possible.

Critical Considerations of the Direct Recovery System.—

Having now described the principal types of plant used in the direct recovery process, it will be useful to add a few critical notes on actual working.

A writer in *The Gas World* (By-Products Section) of March 6th, 1915, says: "Pamphlets, catalogues and trade publications in general would convey the idea that direct recovery of ammonia is an ideal process, never beset by any of the difficulties and objections which are offered against the old indirect process. In the writer's mind there is no question that the direct process is far superior, taken all round, to the indirect; but even in the case of direct recovery difficulties do crop up, and times arise when the working of the process causes no small anxiety to the man in charge. Really, the so-called direct processes consist of two essentially different systems. In one, as exemplified by the Otto process, the tar is extracted from the gas while hot, and no ammoniacal liquor is produced for distillation. In the other type—such as the Mont Cenis and Koppers processes—the gas is cooled before the tar is extracted, and ammoniacal liquor is produced, which has subsequently to be distilled. . . . Temperature of the gas is one of the most important factors in the successful operation of direct recovery processes. In the high temperature processes the gas enters the saturator at about 80° C., the tar being removed at a slightly higher temperature than this. A varying temperature makes it more difficult to regulate the tar extrac-

tion apparatus and to ensure the complete elimination of the tar. It also causes variation in the saturation point of the acid bath with regard to the sulphate content, and if excessive condensation is brought about there is a possibility of having an excess of liquor to deal with—a very troublesome matter at times. With low temperature processes also a fairly constant temperature is necessary. If the temperature falls too low, the tar becomes thick, and causes excessive back pressure in the tar extraction apparatus. By regulation of the coolers this difficulty can usually be overcome. It is also helpful to pass steam into the tar extractors along with the gas, thereby raising the temperature of the tar, and bringing about a diminution in its viscosity. Tar extractors should also be frequently flushed with ammoniacal liquor, and, if possible, with benzol occasionally. If the tar extraction apparatus is inefficient, this is soon made evident in the character of the salt, both in the direct and the semi-direct processes. If the salt is contaminated with tarry matter, even though this is not discernible at first, it will ultimately assume a yellowish tint. Such discoloration must not be confused with that due to the presence of arsenic in the sulphuric acid. To make sure of this, the acid should be tested for arsenic occasionally. In the semi-direct processes, the temperature of the gas entering the saturator varies from 40° to 70° C., according to whether it is preheated or not. It would appear that it is not essential to preheat the gas before the saturator in order to prevent condensation. The reaction between ammonia and sulphuric acid is exothermic, and the heat produced thereby should be quite sufficient to balance radiation and other losses, and prevent a fall in temperature of the gas as it passes through the saturator. If the temperature does fall, it means that the outgoing gas is leaving moisture behind it, thereby weakening the bath, and if this is allowed to continue, there will be a gradual dilution of the bath until a point is reached when “salting” will not take place. The object of cooling the gas in the semi-direct processes is to prevent this; but by the cooling, ammoniacal liquor is produced, and so distillation is necessary, with consequent production of effluent liquor. In the hot or direct processes there is no cooling, and no production of ammoniacal liquor, and therefore no distillation. At the same time, the liquor—though not actually produced as such—is there, in the gas in vapour form. It has therefore to be carried right through the saturator, and thus it is especially necessary that the temperature should be carefully controlled and not allowed to fall; otherwise liquor will be deposited, and will have to be dealt with. This must be carefully watched all along the line after the gas leaves the ovens, as excessive cooling at any point will bring down the liquor. Various devices have been resorted to in

order to deal with possible troubles of this kind. Steam is sometimes introduced into the gas at the exhausters—*i.e.*, after the tar has been extracted—with the idea of keeping the temperature up. In other cases a heater is provided, in which the liquor from the saturator can be evaporated, and any deposition of moisture counterbalanced. For this purpose a steam coil is sometimes introduced into the saturator seal-pot, but, on account of the heavy wear and tear on the coil, this device proves somewhat difficult and expensive in operation. Perhaps the best and most practicable method for dealing with the troublesome liquor problem is to manufacture ammonium chloride. When this is done, it is immaterial if some condensation does take place. The liquor so obtained is heated, and the free ammonia driven off into the main gas stream, whilst the fixed ammonia, consisting principally of ammonium chloride, is obtained as a concentrated solution. By the addition of a little hydrochloric acid a fairly pure commercial ammonium chloride can be obtained in crystalline form by further evaporation. This plan has been adopted with fairly successful results on several installations, and in the writer's opinion it is by far the best method of overcoming the chief objection to the high-temperature or direct process."

In an address to the students of the Sheffield University Gas and Coke Oven Students' Association, December meeting, 1914 (*Gas World*, January 2nd, 1914), Mr. E. M. Myers, in dealing with "Direct and Indirect Recovery Systems," observed that in any plant the success of a direct or indirect process was made or marred in the carbonizing department. The difference between the direct and semi-direct processes was that the tar in the semi-direct was recovered as in the old indirect system, by cooling, together with some form of tar extractor—this involving reheating the gas—whilst in the indirect the tar was taken from the gas without any cooling. The tar obtained by the direct systems was very heavy, as higher boiling constituents alone were removed, and it contained little naphthalene. The naphthalene condensed after the saturator, on cooling the gas for benzol recovery, with the result that the coolers got blocked up, unless special means were adopted. One method was to cool the gases suddenly by passing them up towers down which a spray of cold water was passing, and thus extracting the naphthalene. In another system a duplicate cooler was arranged, so that blocks could be cleared at intervals. With regard to the recovery of ammonia, he pointed out that both the indirect and semi-direct systems afford instances of uneconomical working. The liquor contained a large proportion of ammonium chloride, which was more valuable than sulphate, and which, moreover, did not require the

use of acid to convert it into a marketable salt. This was split up by distillation with lime, and then converted into sulphate, but he was decidedly of opinion that it should be recovered independently. The ammonium chloride caused some difficulty in the direct process. In some systems it was allowed to pass forward with the gas into the saturator, where it was decomposed, with the liberation of hydrochloric acid, which had a very deleterious effect on the saturator linings. In other systems it was recovered from the hot gases by washing with water, which was used over and over again until the solution became strong enough for further treatment. This treatment removed the chlorides effectually, and the quality of the sulphate produced in both direct and indirect systems was good. Back pressure was to be avoided, as any alteration of pressure in any part of the plant affected the whole, and so upset the working of the ovens. One of the most prolific sources of this back pressure was the saturator, where it arose from the blocking up of some part of the apparatus—the curtain, slots, the acid separator and the space between the curtain and the saturator lining being all sources of trouble at times. He advocated making up the bath after each batch of salt, as a means of preventing blockage. To prevent pitching up the hydraulic main, tar must be kept in circulation. In the indirect and semi-direct types of plant this offered no difficulty, as the tar was of a light fluid nature, but in the direct systems it was of a heavy character, semi-solid at ordinary temperatures, and if tar of this kind was circulated for any length of time it became thicker still, and rapidly lost its power of sweeping out any pitch deposits. In addition, it was highly contaminated with ammonium chloride, and if allowed to cool would exhibit small grains of chloride on the surface. If allowed to stand in the mains and pipes, also, by reason of stoppage of the pump, it would set. He had found it necessary to mix part of the oil from the condensers with the tar in order to make it thinner. The tar from direct plants contained a large quantity of ammonium chloride, and must be washed with steam before distillation to recover this, as the chloride caused considerable corrosion in the tar still, and blocked up the condenser. This difficulty was also present in semi-direct processes, but not to such a large extent. Besides the blockages in the hydraulic main and saturator, trouble might also be experienced with naphthalene blockages occurring after the saturator. Unless the naphthalene was removed, deposition was bound to occur, and this necessitated shutting down and steaming out. Each system had certain advantages and disadvantages. In the indirect process large quantities of liquor were produced, giving rise to losses in ammonia, and this liquor had

to be distilled, and the effluent dealt with. The claim that direct systems produced no effluent liquor was not altogether accurate, as moisture contained in the coal charged into the ovens was taken out in the condensers. This effluent was very useful, however, in the condensers. One way of using it was to allow it to trickle over pipes already incrustated with water deposit, both organic and inorganic. The results obtained were surprising, as in the course of time, varying from a week to a month, according to the thickness of the incrustation, the whole of the solid matter disappeared. The main advantage of the indirect system was that the saturator was an independent unit. The disadvantages of the semi-direct system were distillation of liquor, effluent liquor, and the fact that the gas had to be cooled down and subsequently reheated. Moreover, if a shortage of acid occurred the ammonia in the gas was all wasted, and the saturator had a tendency to block up. The advantages were:—(1) Elimination of the naphthalene difficulty; (2) production of tar tolerably free from ammonia; (3) the exhausters worked with cold gas. The direct system had also its disadvantages—such as blocked saturators and condensers, the tar trouble mentioned previously, and the need of extra power required for exhausting, as hot gas had to be worked with. On the other hand, at least one man per shift less was required on a direct plant than on the other two systems, no steam or lime was needed for liquor distillation, the effluent obtained could be easily handled, no liquor was produced, and ammonium chloride could be easily manufactured. On the whole, he concluded that none of the systems could claim any advantage over the others, and again emphasized the view that the type of plant installed must be governed by the class of coal to be dealt with.

Turning to the question of the efficient working of a direct recovery plant, Mr. Myers continued, "I will enumerate several points which I think will tend to be helpful for the guidance of those who will have under their care or notice the arranging of such a plant. The sulphate plant should be as near to the ovens as possible, so as to avoid long lengths of main, and the tar cleaning pockets should be of ample proportions. The exhausters should be steam driven, either of the rotary Beale type or turbo-exhausters. I emphasize the point of steam driven exhausters because I think they tend to more flexible working, and are less liable to stoppages due to failure of current—a fact which is very important on large coking installations. Even in the best regulated power houses something occasionally goes wrong, and my experience of electricians is that although they may be excellent men in their profession, they are not always able to ensure continuous running.

Special attention should be paid to the draining of the plant, and wherever oil, tar or liquor tanks are fixed they should be surrounded with suitable trenches, lined with blue bricks, leading into a common tank. This is also applicable round the benzol scrubbers, so that if leakages of oil do occur no apprehension need be felt as to loss. Seal pots should be of ample capacity, to allow of easy cleaning with ladles, and should be so arranged that if they 'blow' the oil or tar or liquor which they contain will be led away to the common tank. Where pumps are installed, direct-acting steam pumps, or electrically driven turbine pumps, should be stipulated for, and they should be erected on beds fitted with drains, so that in the event of examination of the valves or packing, all leakages are conducted to the common tank. Whilst on the question of pumps, I might say that only first-class steam valves should be used, inferior valves being absolutely useless, and more costly in the long run. Another point in this connection is to insist on first-class piping for pump suction and deliveries, and also to ensure that all joints are perfectly tight, and not simply screwed up and then caulked if they leak. When long deliveries are necessary the pipes should be securely clamped, so that no vibration can cause the joints to become slack and leak. It is a mistake to use common piping on a direct recovery plant, because the presence of ammonium chloride is not at all conducive to long life. Where pipes are laid in the ground, suitable trenches should be provided, made of brickwork and covered with plates. Nothing is worse than to find joints leaking underground, and having to disturb paving, etc., and not being quite sure as to where the pipe is. Ample use of flanges should also be insisted on, so that when piping has to be renewed it can be done with a minimum of labour and cost. A great fault of coke oven constructors is to erect a plant with the idea that it should never come to pieces, and sometimes the greatest difficulty is met with in renewals. Steam supply pipes should be properly covered with non-conducting material, and steam should be laid on to all points requiring the removal of naphthalene. All the mains should have machine faced flanges to ensure a tight joint, and expansion joints should be inserted where necessary. Pressure gauges, preferably of the 'dead beat' type, should be fitted at every available spot, so that the pressures can be read at a glance. The installation of a central battery gauge board in the exhauster house looks nice, but is not at all useful. Valves must be relied on to close tight without any undue forcing, and I have found that a suitable type is the Bryan Donkin design. It is important to see that the whole of the valves on the plant are periodically tried, so as to ensure their being in thorough

working order. Lead acid valves and mother liquor injectors installed in the sulphate house should be of an approved type, and of such a design that they will have a maximum life. The saturators should be designed so as to allow of easy inspection. Manhole lids should be provided wherever possible, so that, when necessary, the examination of the saturator can be made periodically with comparative ease."

Liquid Ammonia Plant.—Where the ammoniacal liquor is not sold or the ammonia recovered as sulphate, it is usually worked up as concentrated gas liquor, or as liquid ammonia. Concentrated gas liquor is generally of two strengths: (1) containing 16 to 18% of ammonia, with sulphide and carbonate present, and (2) containing 18 to 26% of ammonia, with some sulphide, but little or no carbonate. In (1) the gases from the sulphate stills pass through a reflux condenser, which removes some of the steam, and afterwards through a direct condenser, the vapour being then absorbed in water. In (2) the gases from the reflux condenser pass through a milk of lime washer, which absorbs more of the carbon dioxide and some of the sulphuretted hydrogen, and are then absorbed in water as in (1).

In the production of pure aqueous ammonia, excess of lime is necessary to remove all the acid contents of the liquor, including carbon dioxide and sulphuretted hydrogen. The liquor is usually preheated, distilled as in sulphate making, the gases passed through a reflux condenser, and then through two or three cream of lime washers for the absorption of carbon dioxide and sulphuretted hydrogen, ferrous sulphate being sometimes added to the last lime washer to take out the last traces of the H_2S ; solutions of sodium permanganate or ammonia persulphate also being used for this purpose. The vapours are then passed through charcoal scrubbers, and sometimes through a further scrubber containing a fatty or high-boiling mineral oil, after which they are absorbed in water. (Thorpe's Dictionary of Applied Chemistry, Vol. I., 1912 edition, p. 150.)

Fig. 39 is a diagrammatic sketch of the liquid ammonia plant installed at the Greenock Gas Works, which the author is enabled to reproduce by the courtesy of the manager, Mr. James Macleod, F.I.C., F.C.S. A is the overhead liquor storage tank, B is the liquor meter, C contains the coil heater, D is the primary still, E is the pump for pumping the liquor from D through F back to G. F is the liming vessel, G is the main still, H is the reflux condenser, I is the water tube condenser, J is the atmospheric condenser, K is the soda washer, L is the oil washer, M is the permanganate washer, and there is another vessel similar to M used as a second oil washer. N is an absorption tank, and

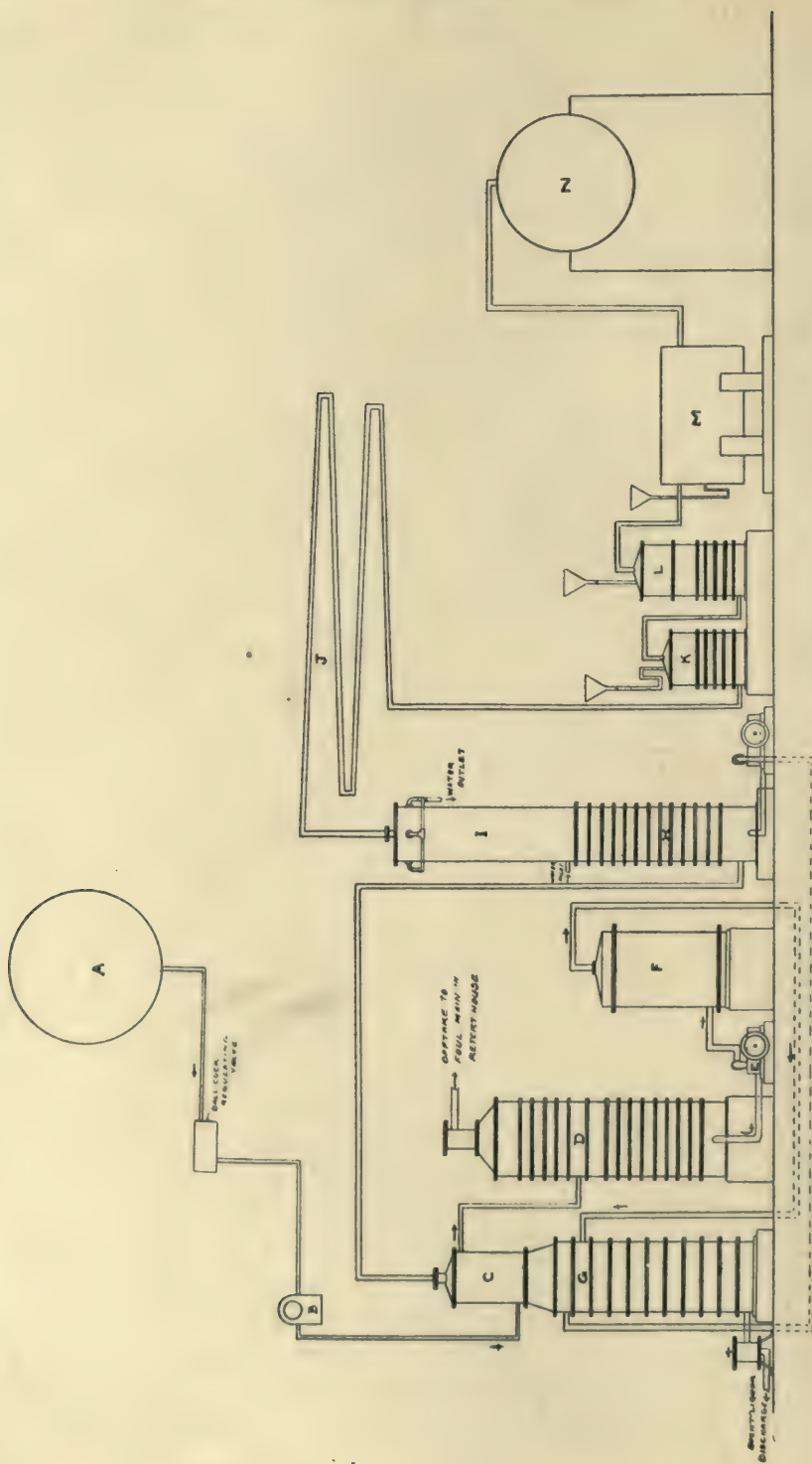


FIG. 39.—DIAGRAMMATIC SKETCH OF LIQUID AMMONIA PLANT AT THE GREENOCK GAS WORKS.

there are in all three tanks similar to N. The extra washing arrangements in this plant are due to the fact that an ammonia solution must be produced which will give an absolutely colourless bicarbonate of ammonia, for the manufacture of which it is used.

Fig. 40 shows the plant of the Gas Machinery Company, Cleveland, Ohio, U.S.A., for the manufacture of aqua ammonia from crude ammoniacal liquor. Mr. P. Plantinga (*Gas Record*, Chicago, Jan. 22nd, 1916, and *Journal of Gas Lighting*, Feb. 1st, 1916) points out that if aqua ammonia is to be made on a large scale, the process must not only be continuous, but the apparatus must necessarily operate with a minimum of purifying agents and expense. The apparatus described requires (exclusive of the lime used for recovering the fixed ammonia contained in the crude liquor) only an exceptionally small quantity of caustic soda, oil, charcoal, and lamp black for the elimination of such impurities as cannot be automatically driven off by the apparatus itself.

The illustration shows the general design of the apparatus, in which the automatic elimination of the impurities from the liquor is based on the fact that carbonic acid and sulphuretted hydrogen are easily driven off in large quantities when crude liquor is heated to (or nearly to) its boiling point.

The machine operates as follows:—The cold crude gas-works or coke-oven liquor is pumped into an overhead feed tank, from which it flows at a uniform rate into the top of the preheater. While passing down through the preheater it is heated by live or exhaust steam, which drives off the larger portion of the volatile impurities—such as carbonic acid and sulphuretted hydrogen. Some ammonia also is driven off, but this ammonia is caught in the upper (washing and cooling) sections of the preheater by the cold crude liquor. Any ammonia vapours passing through these sections are arrested in the small scrubber placed on the top of the preheater, by the small amount of fresh water which flows through the scrubber. The volatile impurities—such as the carbonic acid and the sulphuretted hydrogen—pass from the scrubber into the atmosphere. The hot treated crude liquor, freed in the preheater from a very large portion of the volatile impurities, flows into the top of the ammonia still. The ammonia is eliminated from the crude liquor in the still by passing live or exhaust steam into the bottom of the still. After the volatile ammonia is driven off in the volatile still, milk of lime is added in the liming leg, to free the fixed ammonia. The waste liquid running out at the bottom of the still only contains an exceedingly slight trace of ammonia. The ammonia and steam vapours leaving the volatile still enter a water-cooled condenser placed on the top of the still; and

a considerable amount of the water vapour is eliminated in this condenser—the condensation flowing back into the top of the still. The purification of the ammonia vapours is mainly accomplished in the water washer. A small stream of fresh water is run continuously into the top of this washer, and forms aqua ammonia on its way down. The ammonia in the water washer combines with the remaining carbonic acid and sulphuretted hydrogen contained in the ammonia vapours, and in this way

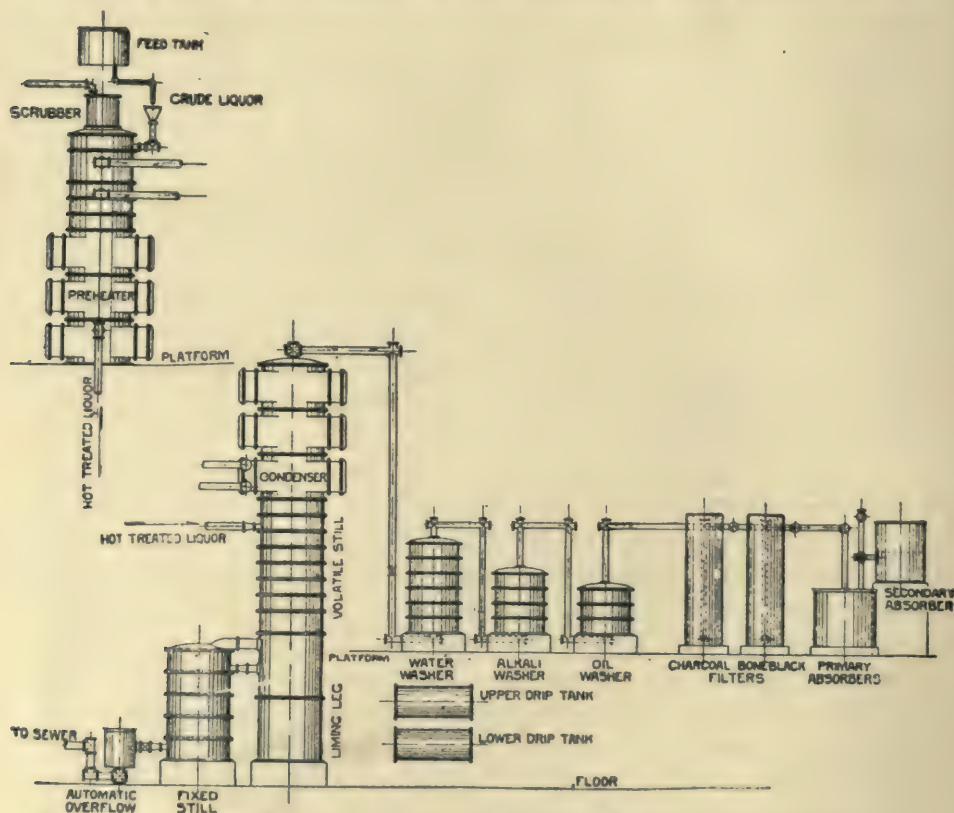


FIG. 40.—LIQUID AMMONIA PLANT, GAS MACHINERY CO., CLEVELAND, OHIO.

these impurities are entirely eliminated. As considerable heat is evolved by the absorption of ammonia in the water, the water-washer sections contain water-cooled coils. The overflow from the water-washer runs into the upper drip-tank, from which it is drained periodically into the lower drip-tank. From the latter tank it is usually run back to the crude liquor tank or well, or the drippage is forced by compressed air into the preheater, where the larger portion of the volatile impurities is again driven off.

The caustic or alkali washer is used for a safety or catch washer ;

a small amount of caustic soda solution being added periodically to arrest such impurities as may pass the water-washer accidentally. Only an exceedingly small amount of caustic soda is required. The oil washer is used for eliminating organic compounds, and fresh oil is seldom added. Two filters, one filled with charcoal and one with bone-black, are used in series to remove the colouring matter from the ammonia gas. The charcoal and bone-black are seldom renewed. If the plant is to be operated continuously, two pairs of filters are installed with the required by-pass connections to allow of one pair being kept in reserve.

There are two primary absorbers for the purified ammonia gas. While one is absorbing ammonia to make aqua ammonia, the other is emptied, and then filled with distilled water. To prevent any waste of ammonia, a two-compartment secondary absorber is provided, and the weak aqua ammonia formed in this absorber is drained periodically into the empty primary absorber. The absorbers are provided with cooling coils, as a great deal of heat is generated by the absorption of ammonia in water. The aqua ammonia from the primary absorbers is usually temporarily stored in an aqua ammonia storage tank, and, after being filtered, it is loaded into tank cars, drums, or carboys. The finished product is a water-white aqua ammonia, which not only fulfils the standard specification, but impurities are practically absent. It is, however, not chemically pure, as the aqua ammonia comes in contact with steel in the filters, absorbers, piping, and storage tank.

Plate VII shows an arrangement described by R. W. Hilgenstock in the *Chemical Engineer*, Chicago, (see also *Chemical Engineering and the Works Chemist*, Vol. II., pp. 137-139 and 201-203), for the manufacture of aqua ammonia and sal-ammoniac direct from raw gas liquor. The raw gas liquor is pumped into the overhead feed regulating tank (6) by pump (19), from where it flows into the upper section of the preheater or carbonic acid extractor (1). The three upper rings of this apparatus are provided with lead cooling coils in order to keep the liquor cold, so that it can absorb again any particles of ammonia that should be driven off from the heating chambers below. The liquor travels down from section to section; before it enters the upper rectangular treating chamber, however, it is brought into the lower section of the cooler (3), where it is supposed to cool the ammonia vapours from the still (2), but at the same time is heated up to about 80° C., and goes back now in the upper heating chamber of the apparatus (1). In the three heating chambers the liquor is gradually heated up to about 100° C.

The liquor, after leaving the lower heating chamber of apparatus (1) enters the top section of the still (2), travels down section for section,

and coming in contact with always hotter steam on its way downwards ; the last particles of carbonic acid, sulphuretted hydrogen, and the whole "volatile ammonia" are set free in these upper sections. To recover, also, the ammonia compounds which are not decomposed by heat, milk of lime from apparatus (17) is pumped into the high vessel below these sections, and thoroughly mixed with the liquor by steam. This mixture of milk of lime and gas liquor then flows into the secondary part of the still (2), where it is boiled up again with live steam, to drive off the last particles of ammonia. The waste liquor leaving this part of the still does not contain more than 0.005% NH_3 . The sections of the secondary still and those of the primary are entirely the same.

For the settling of the lime in the liquor, large settling-basins, made in heavy concrete, are provided, and the overflows are arranged so that the liquor has to travel a long way in order to give the lime ample time to settle ; at the end of this purifying plant the liquor is absolutely clear, so that it can be piped into the city sewer.

Returning to the still (2). The ammonia vapours, containing also carbonic acid and sulphuretted hydrogen, which are set free in the different sections of the still, travel upwards into the cooler (3) of special construction, which serves the purpose of retaining most of the steam vapours that are carried up by the ammonia, the condensed steam vapours flow back into the upper section of the still, while the ammonia gases leave the cooler comparatively dry, but still contain a certain percentage of carbonic acid and sulphuretted hydrogen. To remove these impurities, the ammonia gases are washed with aqua ammonia in a so-called water-washer (9) ; care, however, must be taken that only a certain quantity of water is brought into this apparatus ; it must be sufficient to absorb enough ammonia for retaining carbonic acid and sulphuretted hydrogen.

To be absolutely sure that even in irregular operation no carbonic acid can pass into the filters, which would also mean stoppage of the pipes and fittings, a further smaller washer (10) is provided, filled with a solution containing about 20% of caustic soda ; carbonic acid and sulphuretted hydrogen would combine with the soda, forming carbonate of soda or sodium sulphide ; both compounds are soluble in water. At the same time, however, the washer serves the purpose of further drying the ammonia gases, as some of the moisture which is carried mechanically along will be retained by the soda. To relieve the gas from oily and tarry substances, an oil washer (11) is installed, which is filled with a heavy mineral or paraffin oil.

CHAPTER II.

BY-PRODUCT RECOVERY COKE OVENS.

The adoption of the by-product recovery oven in British coking practice has made very considerable headway, but much still remains to be done in this direction. In an address before the Manchester meeting of the British Association in 1915, Professor W. A. Bone estimated that if all the coke required in this country were produced in by-product ovens, taking into account the relatively greater yield of coke from a given weight of coal, as well as the value of the by-products, the saving effected would not be less than £2,000,000 yearly. In a paper read before the Junior Institution of Engineers, Mr. G. S. Cooper stated that in 1911 the weight of coke manufactured in this country other than at gas-works was 11,474,174 tons, representing a coal consumption of nearly 20,000,000 tons, and approximately half was made in beehive ovens without recovery of the by-products.

In the last-mentioned paper, Mr. Cooper (*Times Engineering Supplement* report) has the following notes on the subject:—

The manufacture of coke for metallurgical purposes began early in the 17th century, but it was not used to any extent until the introduction of the blast furnace, when it was realized that coke was a better fuel than charcoal. Before 1861 the only oven in use was the beehive type, and with such ovens none of the volatile constituents were recovered. In 1861 the Coppée retort oven was introduced, and this was the forerunner of the modern by-product recovery oven.

Coke made in by-product ovens at first met with opposition, and for a long time many ironmasters would not use it for furnace work. The experiments made on a practical scale by Lowthian Bell did much to remove this prejudice. In Germany there is practically no beehive coke to-day. In the United States of America an enormous amount

of beehive coke is manufactured, but considerable attention is being paid to the by-product process, and the United States Steel Corporation has erected a large number of by-product ovens.

With the majority of beehive ovens coke is the only product, but with by-product ovens are obtained coke-tar, sulphate of ammonia, and benzol, together with a large quantity of illuminating gas. A typical South Yorkshire coking coal, carbonized in beehive ovens, would yield about 60% of coke. In by-product ovens the yield of coke would be at least 70%, and the by-products obtained would be approximately 5% of tar, 1.5% of sulphate of ammonia, 1.0% of benzol, and 5,500 cubic feet of surplus gas per ton of coal, assuming regenerative ovens were used. With the increasing use of tar for road-making purposes, of sulphate of ammonia as a fertilizer, and of benzol and toluol, it is evident that the country is losing heavily by neglecting to recover all these valuable constituents of coal.

Practically all the modern by-product recovery ovens are direct developments of the old Coppée retort oven, or of the Simon-Carves oven. Two systems of heating the ovens are in general use: (1) by means of vertical flues and (2) by means of horizontal flues. In the case of vertical-flued ovens the heating flues are distributed at equal distance along the oven wall, while in the case of horizontal-flued ovens the flues are super-imposed one above the other to the height of the oven. In order that the charge of coal may be thoroughly carbonized in the shortest possible time, it is essential that the oven should be heated uniformly throughout its whole length, and various arrangements have been adopted to secure such uniformity.

There are two distinct types of oven in general use, *viz.*, the waste-heat oven and the regenerative oven. In the former the heating gas is supplied with cold air for combustion, and the hot waste gases are led away to a range of steam boilers, where the heat is abstracted from the gases. In the regenerative type the air for combustion is pre-heated to a degree in regenerator chambers of the Siemens type. With both types equal results are obtained so far as the quantity and quality of the products is concerned. With a battery of 50 waste-heat ovens carbonizing approximately 300 tons a day, about 320 tons of steam can be raised per day. The usual type of colliery steam engine consumes about 22lb. per b.h.p. hour, therefore the power obtainable is 1,360 b.h.p. per day. With 50 regenerative ovens of the same capacity, since each ton of coal yields approximately 11,000 cubic feet of gas, and 5,500 cubic feet of surplus gas per ton, while large gas engines utilize about 21 cubic feet per b.h.p. hour, the power obtainable is 3,275 b.h.p.

per day. Coke oven gas is especially valuable as a fuel in steel furnaces for ingot heating, steel ladles, etc., and for open hearth furnaces. This outlet for the surplus energy of a coke oven plant is a very valuable one, but can be used only where regenerative ovens are employed.

All coal cannot be converted into coke. Generally speaking, a coal which contains a high percentage of oxygen possesses little coking power, though there are several well-marked exceptions. Probably the best coking coal in the country is that mined in Durham, but excellent coke is also obtained from Yorkshire and South Wales coals. The average Durham coal is charged into the ovens in a normal state, containing about 2.5% of moisture, and crushed fine. The coal is not compressed, and a good, hard, dense coke is produced. In South Wales the coal is charged in the same way, but has a tendency to swell on being carbonized. In Cumberland still another variation is met with. There the coal contains about 30% of volatile matter, as against 25% in Durham, and 15-20% in South Wales. If it is charged into the oven, as in Durham, the coke produced is soft and friable, and will not stand much handling. In order to produce a harder and denser coke the coal is compressed into the form of a cake containing about 10% of added moisture, and so charged into the oven.

The major portion of the coal used in this country for coking is subjected to a washing operation before being carbonized, so as to reduce the ash content, and eliminate some of the sulphur compounds. After washing it is crushed and deposited in a storage bunker, where the excess of water is drained off. Two methods of charging the crushed coal into the ovens are employed, *viz.*, (1) from above, the coal passing through holes in the roof of the oven, and (2) through a door, the coal being fed into the oven in the form of a compressed cake. Generally speaking, a coal containing above 25% of volatile matter has to be compressed, while coal with less than 25% is charged in the normal state.

The main points of difference between the several systems of ovens are as follows:—(1) The arrangement of the heating flues; (2) the facilities for inspecting all the working parts of the ovens; (3) the method of regulating and controlling the heating of the ovens; (4) the pre-heating of the air required for combustion. The essential features of a good oven are:—The heating must be easy to regulate and control; the temperature should remain the same throughout the whole length of the oven wall, in order to produce a homogeneous coke; ample means for controlling both gas and air supplies should be provided; all working parts should be easily accessible for inspection; the

amount of gas required for heating the ovens should be reduced to the minimum.

The Coppée Regenerative By-Product Oven.—Writing editorially on the Coppée coke oven, *The Gas World* (By-Products Section) said recently :—"Indirectly, as well as directly, the industry owes a great deal to this oven which is not generally recognized. The original Coppée ovens were the first successful retort ovens to be built in this country, and from them the modern vertical-flued retort oven has undoubtedly sprung. The earlier experiments with regard to the proper width of ovens were done on Coppée plants, and, moreover, by their use the pushing machine or ram became necessary. This, in turn, was developed and perfected until it reached its present-day standard. Some thousands of the original Coppée ovens were built in Great Britain, and we certainly owe much to their originator."

The Coppée ovens are shown in Figs. 41 and 42. In the regenerative type of oven the heat in the burnt gases is extracted by passing them through a gallery filled with fire-bricks which absorb the heat during a period of about half-an-hour, and in the following half-hour the air necessary for combustion passes through the heated fire-bricks, where it is raised to about 1,000° C. Owing to this fact, regenerative ovens require only about 50% of the gas evolved to coke their charge of coal, and there is therefore about 50% of the gas available for purposes other than heating the oven. In the Coppée oven the abstraction of this heat is brought about by causing the hot burnt gases to travel through a regenerator which runs under the whole length of the battery of ovens. This arrangement gives the largest possible amount of regenerative surface, which is proved by the low temperature of the gases escaping to the chimney.

The oven is charged by means of aadies or a charging machine, through holes in the top, and levelled by machinery or hand; or where suitable the coal is compressed into a cake, and then pushed into the oven on the peel or cake-plate attached to the charging machine. The volatile products evolved from the coal pass up the vertical ascension pipes into the gas main, and so on to the by-product plant, where they are treated as described in Chapter I (page 62 and Plate V). The by-products having been extracted, the clean gas is returned to the ovens, where it is used for heating the side walls to the necessary temperature required for coking the charge of coal.

The Coppée regenerative oven is illustrated in Fig. 41. The gas returns from the by-product plant by a gas main which divides and runs along both sides of the battery shown at (1) (1), Fig. 41 A. The

gas can be supplied to the fireclay distributing tubes either (3) (3) (3) or (4) (4) (4), Fig. 41 B, according to the position of the 3-way cocks (2)

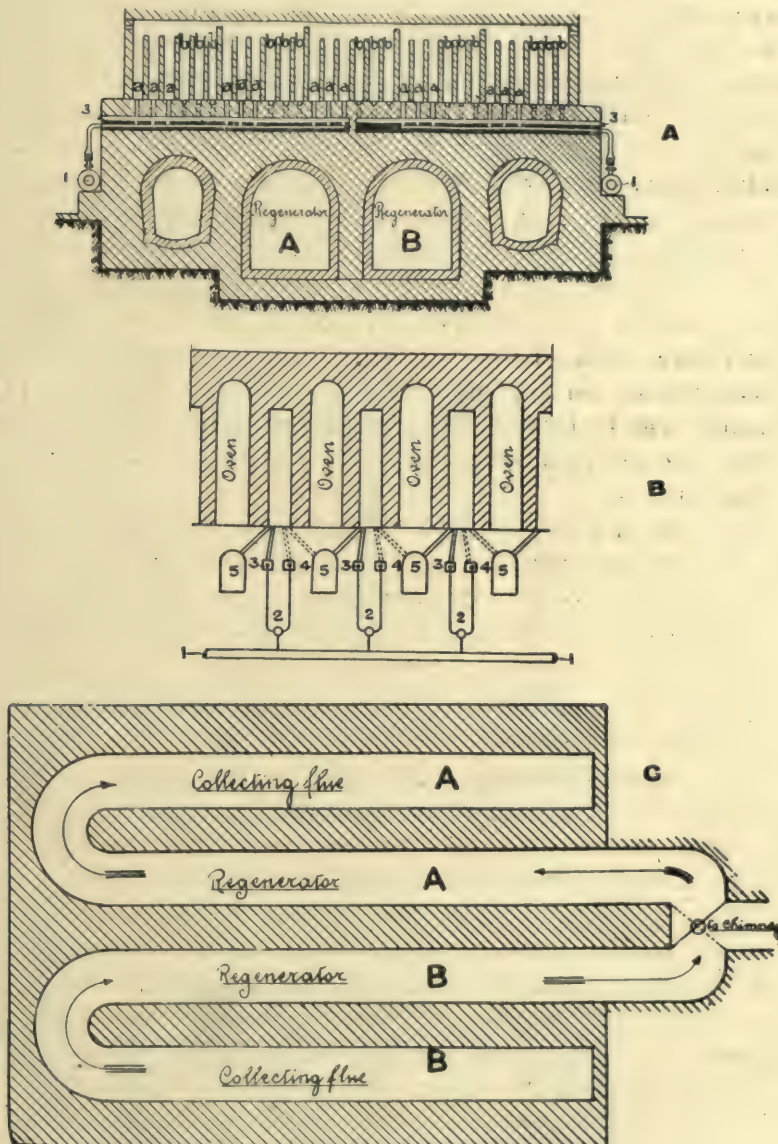


FIG. 41.—COPPEE REGENERATIVE COKE OVEN.

(2) (2). The side wall of the oven is divided into five independent chambers shown in A, and the fireclay distributing tubes (3) (3) (3) admit gas

to the first three flues (a) (a) (a) of each chamber, while the distributing tubes (4) (4) (4) admit gas to the last three flues (b) (b) (b) of each chamber. The air necessary for combustion is supplied by a fan, and its entry to the flues is controlled by dampers situated between the regenerator and the fireclay distributing tubes. When the 3-way cocks are in one position, the mixture of gas and air burns in the flues (a) (a) (a) of each chamber, and the products of combustion pass to the sole flues (5) (5) through the flues (b) (b) (b). At the end of about half-an-hour the 3-way cocks are simultaneously reversed by means of a sliding rod operated by hand, and then the gas and air are reversed so that (b) (b) (b) become ascension flues and (a) (a) (a) are return flues. The flues (a) (a) (a) are connected with one regenerator (say A), Figs. 41 A and C, while the flues (b) (b) (b) are connected with the other regenerator (B). Thus each regenerator in turn is heated for about half-an-hour by the burnt gases, and then for a similar period imparts this heat to the air necessary for combustion. A single lever, which can be operated automatically, serves to reverse the gas and air every half-hour.

This type of oven is capable of carbonizing from 5.8 to 8.6 tons of coal per 24 hours according to the nature of the coal to be coked. The usual dimensions of the ovens are : length, 33 ft. ; height, 8 ft. 3 in. ; and varying in width from 15 in. to 24 in. to suit the class of coal to be coked. Fig. 41 A is a section through the side wall of the oven. Fig. 41 B is a section through the oven and side walls, and Fig. 41 C is a section through the regenerators and collecting flues.

The Coppee Waste Heat Oven.—This oven (Fig. 42) is built in different sizes suitable for coking from 5.2 to 8.5 tons of coal per 24 hours. The gas returns from the by-product plant by a gas main which divides and runs along both sides of the battery shown at (1) (1) Fig. 42 A and C. From here it is led to a mixing apparatus (2) designed on the principle of the Bunsen burner, which allows a regulated quantity of air to enter with the gas into the fireclay distributing tubes (3) (3) which run under the first 12 flues of each half of the side wall. The upper surface of these fireclay tubes is pierced with holes (4) (4) which allow the mixture of gas and air to pass into the vertical flues (5) (5), where it burns and heats the side wall of the oven. By means of the secondary air inlets shown at (1, 2, 3) to (10, 11, 12) in B, a regulated quantity of extra air can be separately admitted to each of the vertical flues. It is therefore easily seen that the dual control afforded by the combination of these secondary air inlets with the primary air inlet at (2) A enables the combustion to be exactly regulated in each individual flue, resulting in the maintenance of an

even temperature along the whole of the oven wall. The products of combustion ascend the vertical flues and reach the top flue (6), from where they descend the down flue (7), and pass through the opening (8)

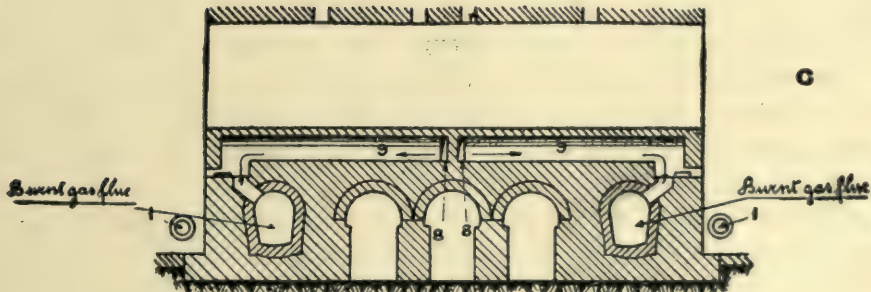
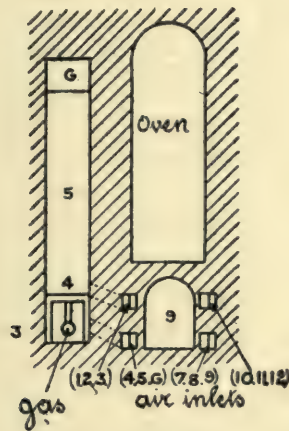
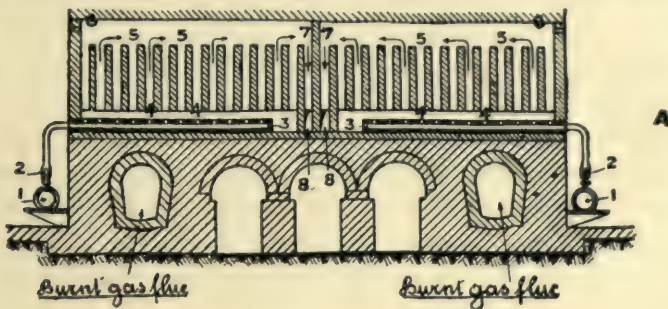


FIG. 42.—COPPER WASTE HEAT COKE OVEN.

into the sole flue shown at (9) B and C, thus heating the bottom of the oven. From here they pass into the collecting flue for burnt gases (C) which leads to the boilers, and finally to the chimney.

An installation of ovens designed and erected by the Coppée Company (Great Britain), Ltd., at the Coedly Colliery of the Welsh Navigation Steam Coal Company, is described in *The Iron and Coal Trades Review* of December 4th, 1914. The ovens, of the regenerative type, are in two batteries of thirty, each oven being 33 ft. long, by 8 ft. 2½ in. high, by 1 ft. 8 in. mean width. The system of heating the ovens is as follows:—In the first place the regenerators run under the whole length of the battery of ovens, thus affording the largest possible amount of regenerative surface. The gas mains are led along both sides of the battery, so that gas is supplied to both sides of the ovens. Each oven side wall is divided into five independent chambers, each of six flues. From the large gas mains two gas pipes are led into each half of each oven side wall. One of these branch pipes admits gas to the first three flues of each chamber, and the other branch pipe admits gas to the second three flues. Each group of three flues serves alternately as ascension flues and return flues respectively with each reversal of the gas, each group being connected to its respective regenerator. Thus each regenerator is in turn heated up for about half-an-hour by the burnt gases, and then for a similar period imparts this heat to the air necessary for combustion. It will be seen that the reversal of the gas affects only one-tenth of the wall of the oven, and thus, owing to the short distance which the hot gases have to travel, the appreciable difference in temperature between the upcast and downcast flues is very small. The oven is thus kept at an even temperature throughout its length. Another important feature in the Coppée oven is the special arrangement of the regenerator, by which the air is made to pass through the whole length of the regenerator, and is thus heated to a uniform temperature before entering the flues of each oven side wall. In most systems of regenerative ovens the air for combustion is heated by passing through the whole or a portion of the regenerator, according to the position of the particular oven in the battery, so that the air supplied to the far end of the battery has become fully heated by passing through the whole of the regenerator, whilst the air supplied to the ovens at the opposite end, having only just entered the regenerator, is only slightly heated. The uniform heating of the air attained in the Coppée system is obviously a most important feature. Each battery of ovens is served by an independent electrically-driven coal compressor, together with a Buchanan coke ram and discharging machine, but the arrangement is such that any one unit can be made to serve either or both batteries. Provision is also made for charging the ovens from the top by means of charging gandies in case of accident to the compressors. The capacity of each

oven is 10 tons, and the time of coking is approximately 32 hours. The battery stack is 171 ft. high from the sole plate of the ovens, and has a diameter of 7 ft. at the top. The coke is discharged on to a sloping bench through a Darby type quencher. The ram travels at the rate of six feet per minute, and quenching water is supplied at 80 lbs. pressure by a Eureka tandem compound pump, which is controlled by a Fisher governor. The water for quenching is taken from the base of the cooling tower, the level in which is maintained from a reservoir on the hill side. The bench of each battery of ovens is provided with catch pits for the interception of the coke breeze. The surplus water, after flowing through a series of settling basins, is returned to the pump sump to be used over again. The gases from the ovens are led into a common main 27½ in. in diameter. This is of exceptional length, so as to take the fullest possible advantage of air-cooling effect, and dips into a pitch cistern for the interception of the condensate. The gases pass through three series of serpentine coolers, which are so disposed that any one unit of each, or the complete cooler, can be cut out for cleaning or repairs without interfering with the continuity. Under each cooler a seal pot is provided for the collection of condensate. The exhausters, and in fact the whole of the auxiliary plant, are installed in duplicate. The exhausters are of the Bamag steam-driven type, running at 70 r.p.m., and each capable of dealing with 265,000 cub. ft. of gas per hour. The gases next pass through one of the two Pelouze and Audouin tar extractors, each of which is capable of dealing with the whole volume of gas. Thence the gas passes in succession through three ammonia scrubbers, a gas dryer, three benzol scrubbers (each 50 ft. high), from these to a further dryer, and thence back to the ovens through a Parkinson and Cowan regulator at a constant pressure of 150 mm. The surplus gas, which amounts to between 50 and 55% of the total gas evolved from the coal, serves to raise steam under four Lancashire boilers, and will be eventually used to fire a further four similar boilers at the Colliery, when the whole of the ovens are put in service.

The scrubbers are carried upon a platform above the apparatus gallery, and the whole of the plant is thus easily accessible for inspection, all pipes being above ground. All the pumps for the scrubbers, etc., are housed in the apparatus gallery, and are of the Eureka steam-driven type. At the end and rear of the scrubbers are three storage tanks each of 30,000 gallons capacity; of these, one serves for condensate and two for ammonia liquor. A fourth tank for the storage of tar has a capacity of 40,000 gallons, and from this the tar gravitates to the loading siding.

Simon-Carves Vertical Flued By-Product Coke Oven, with Regenerators.—The Simon-Carves oven, shown in Figs. 43 to 48, is built on the general lines laid down in the eighties by Hoffmann. The "Hoffmann" oven showed good results for the time, but was later on discarded owing to the impossibility of accurately regulating the course of the flames through the different vertical heating flues, the distribution of these flames being entirely dependent on the sections and areas set out during the construction of the oven, without any possibility of subsequent adjustment. Since then many attempts have been made to regulate the course of the flames through the flues of the regenerative ovens. An important improvement was obtained when, instead of only having one point of combustion and the distribution of the flame throughout the different vertical flues, the gas and air were brought separately at the base of each flue, and only allowed to burn at that point. The difficulty remained, however, to regulate the amount of air and gas distributed at each of these points of combustion. Messrs. Simon-Carves, Ltd., point out that up to the time of taking out their Patent in 1906 it was customary to effect the regulation of air and gas by means of dampers and nozzles arranged within, or closely adjacent to the point of combustion, and that these dampers were moved by rods inserted through openings at either the top, bottom, or sides of the vertical heating flues. In the improved type of Simon-Carves oven the supply of air and gas to each flue is separately regulated from a position outside the oven, with obvious advantages.

The Simon-Carves oven with regenerators is shown in Figs. 43, 44, and 45. The distribution of the gas is shown in Fig. 43 as follows:—The gas is brought in pipe 1 (Fig. 43), and alternately fed by means of gas taps 19 through the distributing boxes 38 and 38a, provided with the necessary taps for the gas channels, 2, 3, 4, 5, and 6 leading into the sections 7, 8, 9, 10, and 11, being correctly proportioned to give the required supply of gas to each set of vertical flues. This gas meets the air at point 13, about the level of the sole of the oven, where combustion takes place.

The distribution of the air is shown in Fig. 44. The air for combustion, after traversing through the regenerator 14 (Fig. 44), rises through the ducts 15 into the distributing chamber 16, feeding the compartments 20, 21, 22, 23, and 24 through the horizontal channels 17 and 18, and two other parallel channels placed alongside, and shown at 29 and 30 on Fig. 45, representing a transverse section through the oven. These compartments correspond with the gas sections 7, 8, 9, 10, and 11, and are connected to them by the openings 13 before mentioned. The supply of air which is allowed to pass along the horizontal channels

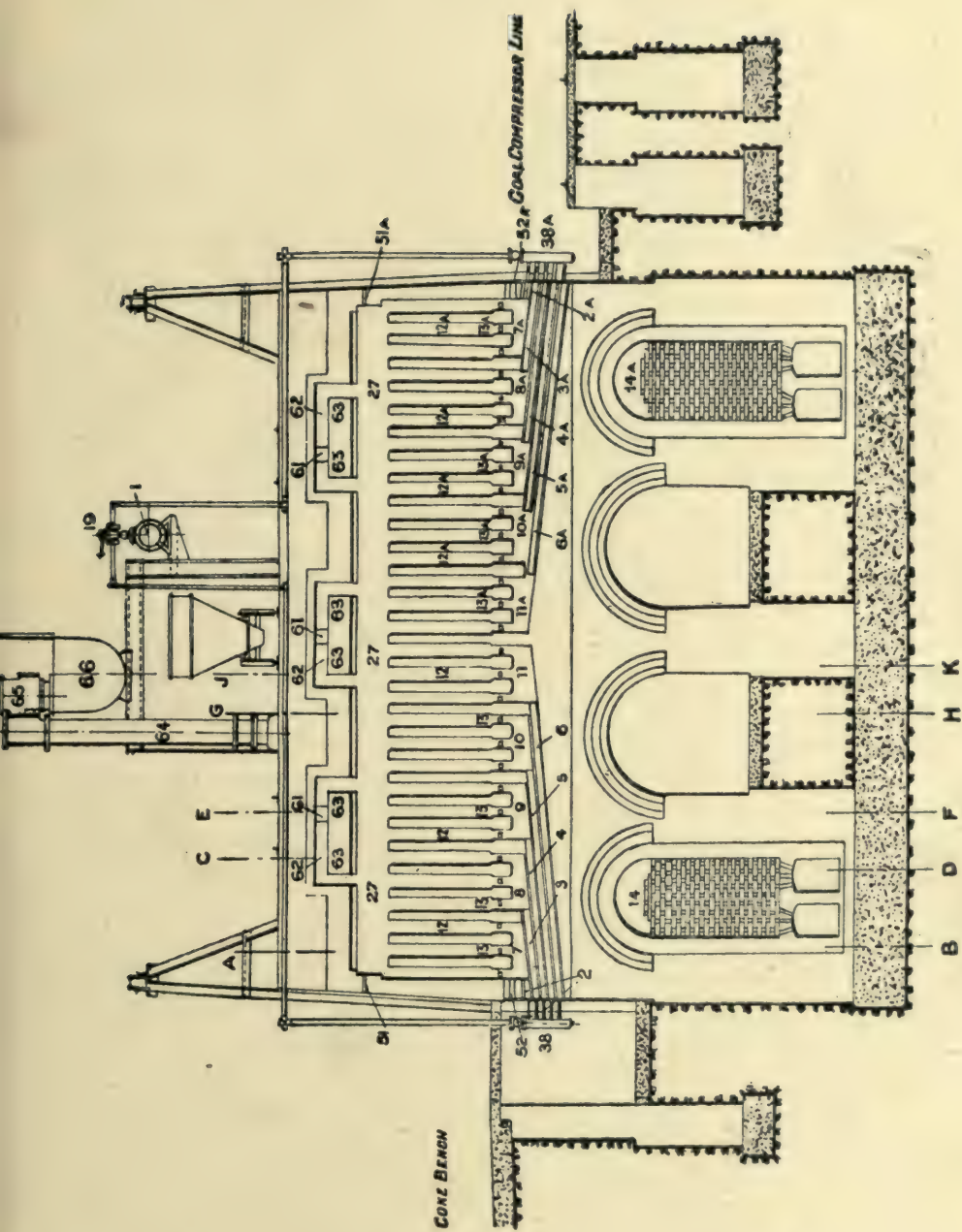


FIG. 43.—SIMON-CARVES BY-PRODUCT COKE OVEN, WITH REGENERATORS, SHOWING DISTRIBUTION OF THE GAS.

is regulated by dampers 25 inserted in these channels, through openings 26 in front of the oven closed with removable plugs. The regulation of the gas, and the air required for its combustion, is thus entirely effected from the outside of the ovens and under the control of the responsible person; and after the final regulation is effected the taps and openings can all be sealed to prevent further tampering.

The flames resulting from the combustion of the gas rise in the vertical flue 12, pass along the horizontal flue 27, and descend the vertical flues 12a, and through the openings 13a into chambers 24a, 23a, 22a, 21a, and 20a, and thence into channels 29a and 30a, and two other parallel channels 17a and 18a placed alongside shown in Fig. 45, leading into chambers 16a and down duct 15a into regenerator 14a, heating the chequered brickwork and passing into the waste gas flue, along which they travel to the chimney. When the chequered brickwork in the regenerator 14a has been sufficiently heated, and the air passing along regenerator 14 has extracted the heat from this regenerator within the prescribed limits, the whole arrangement is reversed, the gas being fed into the sections 7a, 8a, 9a, 10a, and 11a by means of channels 2a, 3a, 4a, 5a, and 6a, as previously described for the other side of the oven, burning with the air at point 13a and rising in the flues 12a, travelling the horizontal flue 27, and descending the vertical flues 12, passing through openings 13 into compartments 20, 21, 22, 23, and 24, and along the channels 17 and 18 and the other two channels 29 and 30 placed alongside, and shown in Fig. 45, into the regenerator 14. The air for the combustion now traverses along regenerator 14a, rising through duct 15a into distributing chamber 16a, and is fed into compartments 20a, 21a, 22a, 23a, and 24a as previously described for the other side of the oven.

The reversing arrangement consists of a set of dampers actuated in pairs on the regenerators, alternately shutting off the waste gas at one end and admitting the air at the other by a winch at the end of the battery. The gas is supplied alternatively to either side of the oven by a set of 3-way gas taps 19, one to each oven, actuated by the same winch.

The great advantage claimed for this type of vertical-flued oven is that in each flue the amount of gas and air admitted for the combustion of the same can be accurately and easily regulated without any inconvenience to the operator. Spy holes being provided at seven points—51, 52, 53, 54, 55, 56 and 57 on each side of the battery—the process of combustion is always under direct observation and control from the outside of the oven. Dampers 59, one at each end of oven, which are

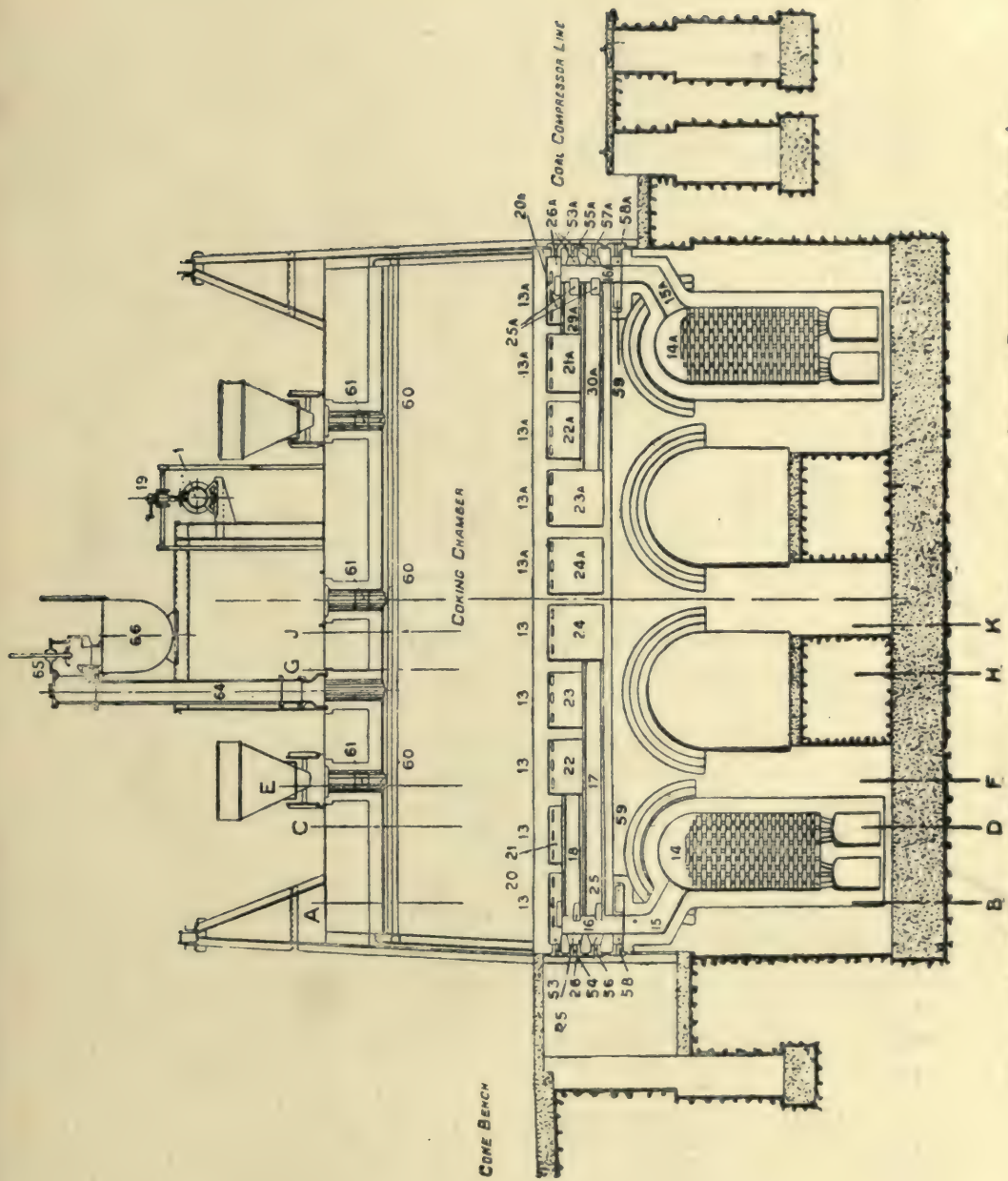


FIG. 44.—SIMON-CARVES BY-PRODUCT OVEN, WITH REGENERATORS, SHOWING DISTRIBUTION OF THE AIR.

regulated through openings 58, control the draught in the oven, and permit shutting down in case of repair.

During the carbonisation of the coal in the oven, the gas evolved ascends through ascension pipe 64, and passes through isolation valve 65 into the main gas collector 66. It is then passed through the cooling and washing plant by the aid of mechanical exhausters, and fed back to the oven through gas pipe 1 as previously described.

For the starting of the oven, fires are provided inside the coking chamber against the two doors.

The hot products of the combustion are collected through apertures 60, and led through openings 61 into channels 62, from which they are distributed by means of ports 63 into the horizontal flue 27 and down the vertical flues 12 and 12a, heating up the brickwork, and escape through the openings 13 and 13a, through the flues under the sole, and down the regenerators 14 and 14a which are then acting as waste gas flues.

When the brickwork of the ovens is hot enough to cause the coal to distil, the openings 61 are closed, and the oven charged with coal, and the gas and air burnt in the oven walls as previously described.

The Simon-Carves Vertical Flued By-Product Coke Oven, without Regenerators.—This oven (Figs. 46, 47, and 48) presents the same features of control of the combustion of the gases, with facilities for the regulation of the heating throughout the whole of the oven, as in the case of the oven with regenerators. The construction of the oven is symmetrical in relation to the centre line, the two halves thus formed being heated independently and similarly.

Under the walls containing the heating flues are situated, side by side at each end of the oven, a set of gas and air channels leading the gas and air separately to the point of combustion, as will be seen from the drawings, which represent a section through the heating wall between two ovens, showing on the left-hand side the gas flues set, and on the right-hand side the air-flues set. The working of the oven is as follows :—

Distribution of the gas: Fig. 46, left-hand side.—The gas is brought in the pipe 1 and fed through the distributing box 2 provided with the necessary taps 3, 4, 5 and 6 to the gas channels 7, 8, 9 and 10 leading into the compartments 11, 12, 13 and 14. The taps 3, 4, 5 and 6 distribute the required amount of gas in each of the flues 7, 8, 9 and 10. The gas, passing through apertures 15, arrives in the combustion

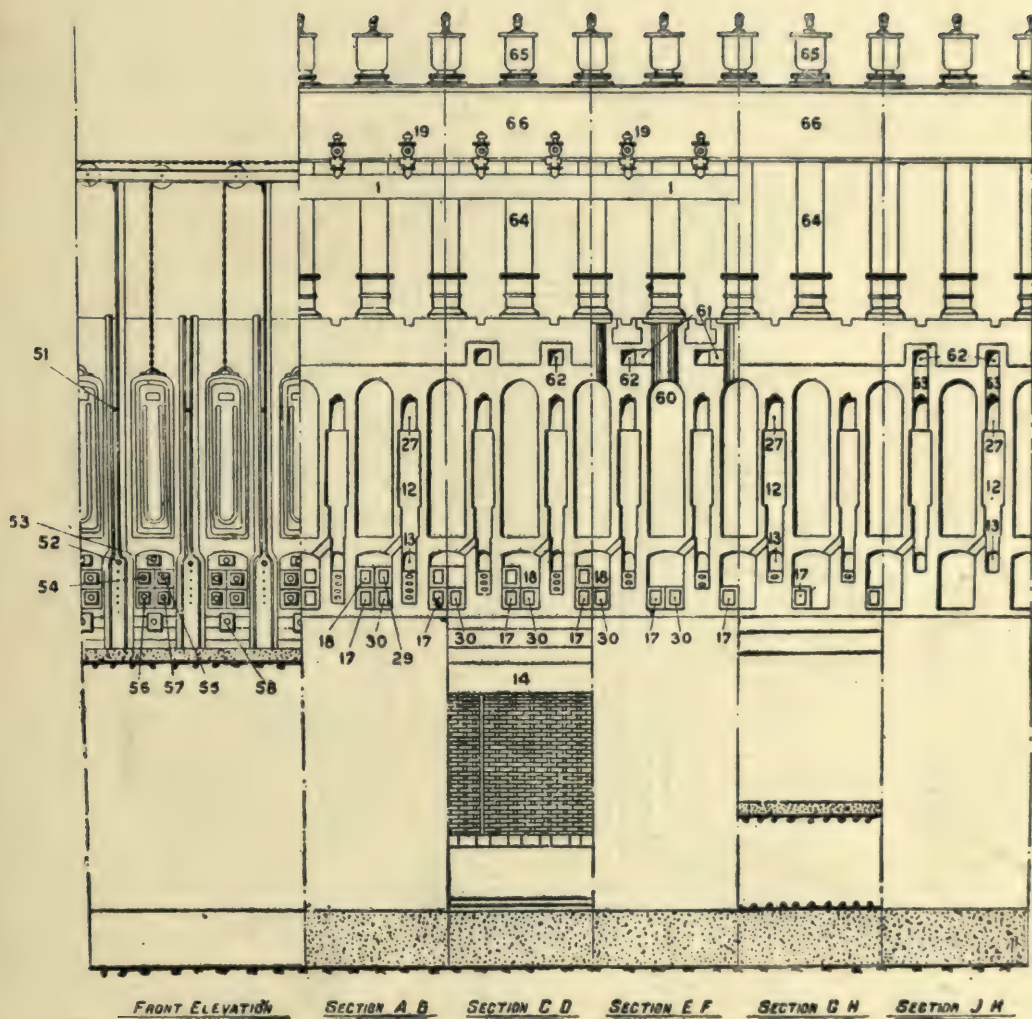


FIG. 45.—SIMON-CARVES BY-PRODUCT OVEN, WITH REGENERATORS; TRANSVERSE SECTION.

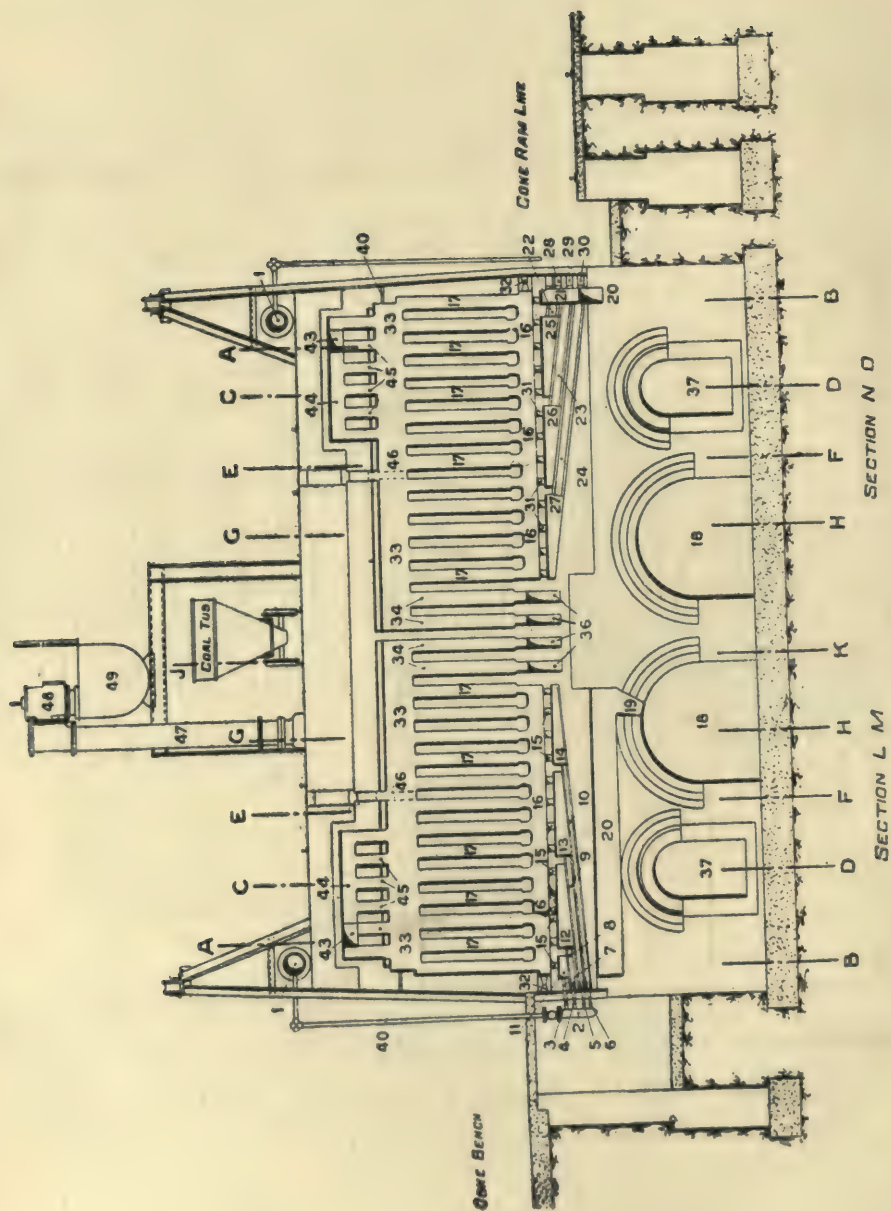


FIG. 46.—SIMON-CARVES BY-PRODUCT OVEN, WITHOUT REGENERATORS, SHOWING DISTRIBUTION OF THE GAS.

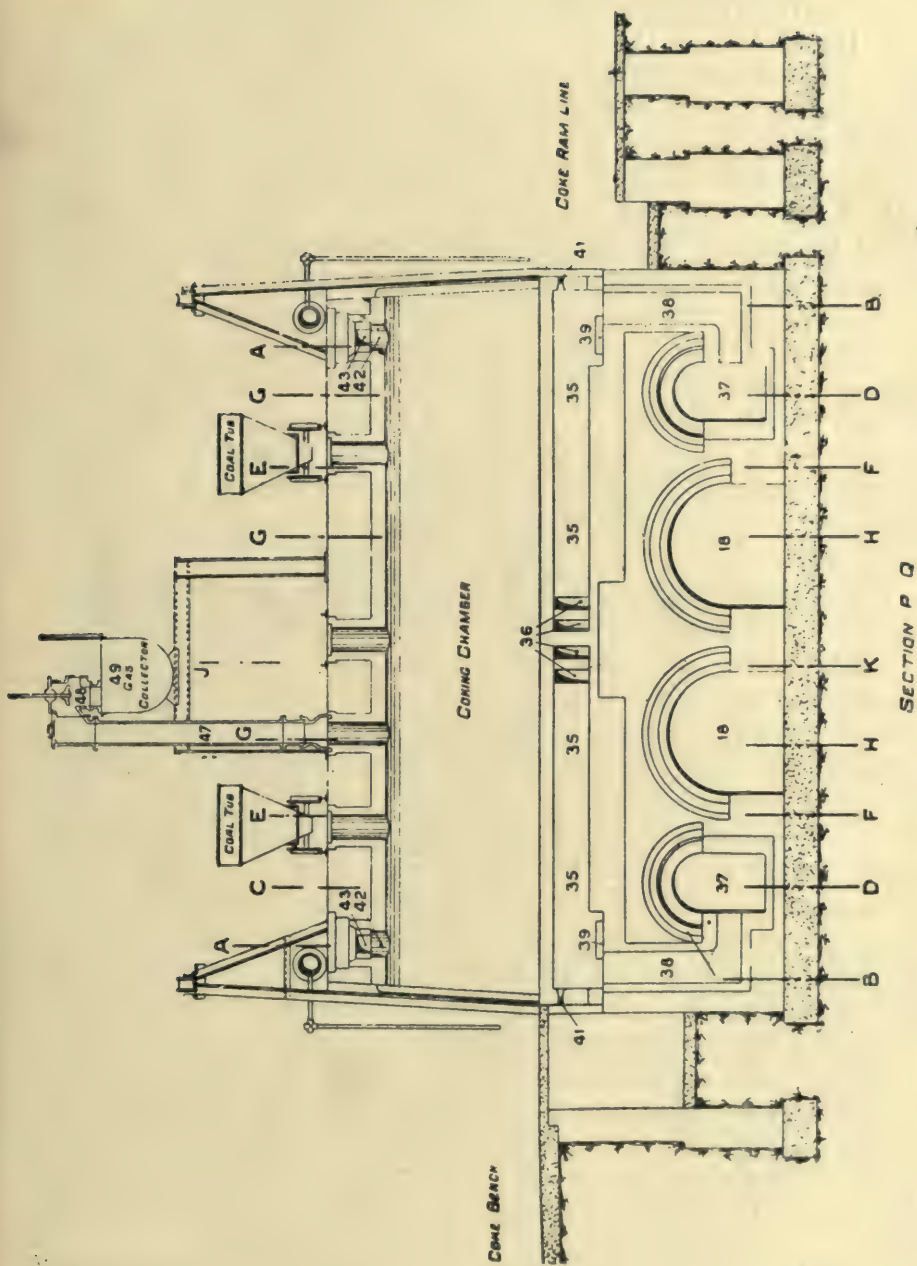


FIG. 47.—SIMON-CARVES BY-PRODUCT OVEN, WITHOUT REGENERATORS, SHOWING DISTRIBUTION OF THE AIR.

chamber 16, situated at the base of the vertical heating flues 17. There is one aperture, 15, under each heating flue 17.

Distribution of the air: Fig. 46, right-hand side.—The air for the combustion, after travelling through the air galleries 18 in the foundations of the battery, where it receives a preliminary heating, passes through the apertures 19, one for each half of each oven, and through the flue 20 into the air distributing chamber 21 represented at the right-hand of the drawing. From this air distributing chamber air passes through the flues 22, 23, and 24 into the compartments 25, 26 and 27 corresponding to the gas compartments 12, 13 and 14, and to the top of the distributing chamber 21, corresponding to gas compartment 11. The amount of air is regulated in each of these compartments by dampers, or stoppers, of different thicknesses, introduced from the outside into the air channels 22, 23 and 24 through the plug holes 28, 29 and 30. The air from each compartment passes through apertures 31 into the combustion chamber 16, where it meets the gas as explained above, and combustion takes place. There is one aperture 31 under the heating flue 17.

Combustion of the gas and heating of the oven.—The whole length of the combustion chamber 16 is visible from the outside through the spy hole 32. The flames resulting from the combustion of the gas rise into flues 17; are collected into the horizontal flue 33 situated at the top of the oven, and then carried down through the centre flues 34 to the flue under the sole of the oven 35 through ports 36, Fig. 47. The sole flue conducts the burnt gases to the waste gas flues 37 (one on each side of the battery) through the downtakes 38. The waste gas flues 37 lead the hot burnt gases to the boilers, where they can be utilised for raising steam on their way to the chimney. The spy holes 32, 40 and 41 on each side of the battery allow for easy inspection through the flues, and permit an accurate control of the combustion of the gases. The dampers 39 (one at each end of the oven) regulate the draught in each oven, and allow of the shutting off in case of repair.

During the carbonisation of the coal charge in the coking chamber the gas evolved ascends through the ascension pipe 47, and passes through the isolating valve 48 into the main gas collector 49. It then passes through the cooling and washing plant with the aid of mechanical exhausters, and is fed back to the oven through gas pipes 1, as described below.

For the starting of the oven, fires are provided inside the coking chamber against the two doors. The hot products of combustion are collected through apertures 42 formed through the arch of the coking chamber, and led through flues 43 and 44, and the distributing ports 45,

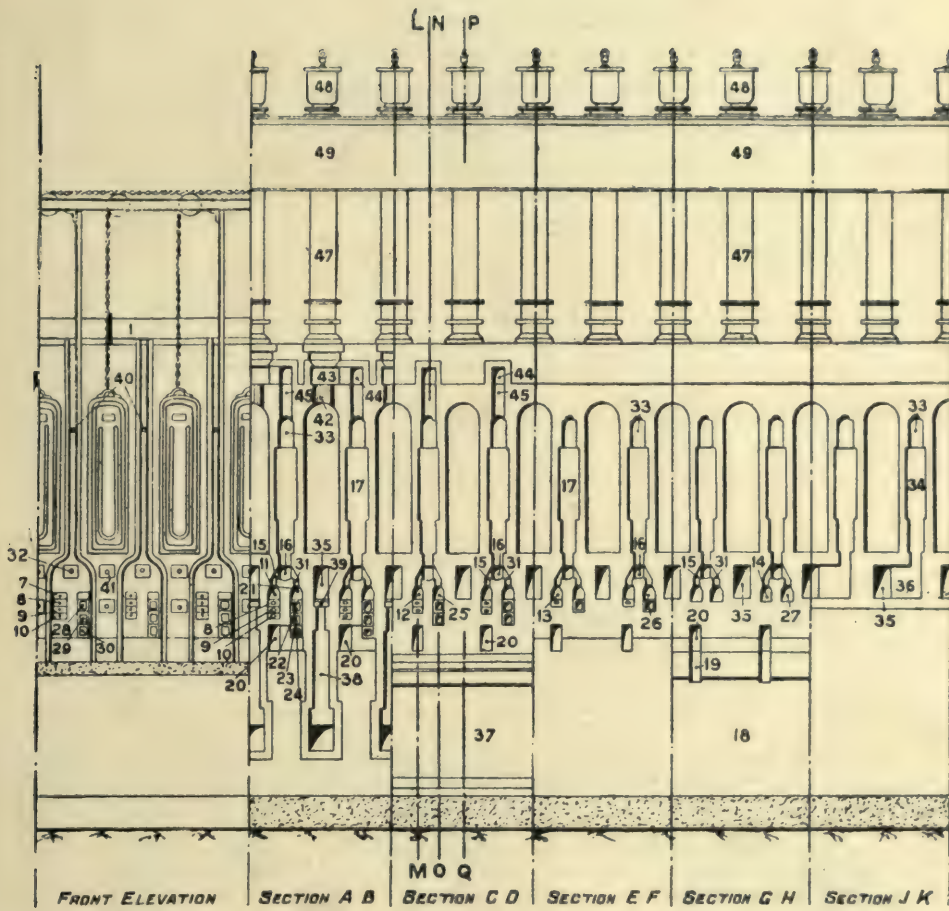


FIG. 48.—SIMON-CARVES BY-PRODUCT OVEN, WITHOUT REGENERATORS, TRANSVERSE SECTION.

into the vertical heating flues of the wall 17. By means of the temporary dampers 46 inserted in horizontal flue 33 the hot products of the combustion from the fires travel down the eight first flues 17, along the combustion chamber 16, rise up through the five next flues 17, and then down the centre flues 34 to the sole flues 35, and thence to the waste gas flues 37. When the brickwork of the oven is hot enough to cause the coal to distil, the openings 42 are closed with a special cover, and the temporary dampers 46 withdrawn; the oven is charged with coal, and the gas and air fed into the oven in the manner described above.

The Koppers' Waste Heat Coke Oven.—The Koppers' waste heat oven is shown in Figs. 49 and 50, which are longitudinal sections through the oven chamber and heating flues. Fig. 49 is a longitudinal section through one of the walls, and shows the arrangement of the heating flues; Fig. 50 is a longitudinal section through the oven chamber; and Fig. 54 shows three different cross-sections through a number of ovens. The oven is a chamber 32 feet 10 inches long, 6 feet 7 inches high, and from 20 to 24 inches wide in the centre. The height and the width, however, vary somewhat according to the class of coal to be coked. The top of the oven is provided with three openings, A, for charging the coal, and a fourth opening, B, through which the gases of distillation are drawn off to the condensing plant. The purified gas is returned from the bye-product plant by the main, C, placed in a position convenient for access, either in front of or at the back of the battery of ovens. Separate branch supply pipes, D, fitted each with a regulating cock, conduct the gas to each oven; and these branch pipes communicate with the gas distributing channel, E, placed immediately below the heating flues. This channel, E, is formed of firebrick pipes, and runs along at the base of the whole of the heating flues, with the exception of one or two at the end remote from that at which the gas is admitted, and these latter flues conduct the waste heat to the main flue, P, leading to the steam boilers. The heating gas passes out of the distributing channel through orifices, each fitted with a gas nozzle communicating with each vertical flue.

The air for combustion flows along the conduit, F, and induced by the chimney draught, it is drawn into the air-distributing channels, G, situated immediately beneath the oven chambers. The air enters each heating flue through the openings, H, and meets the gas issuing through the gas nozzles, U. Combustion takes place a little above the nozzle, the comparatively cold air flowing round the base of the nozzle keeping it cool, and preventing any likelihood of the nozzle setting fast. The products of combustion of the gas and air pass up the heating flues,

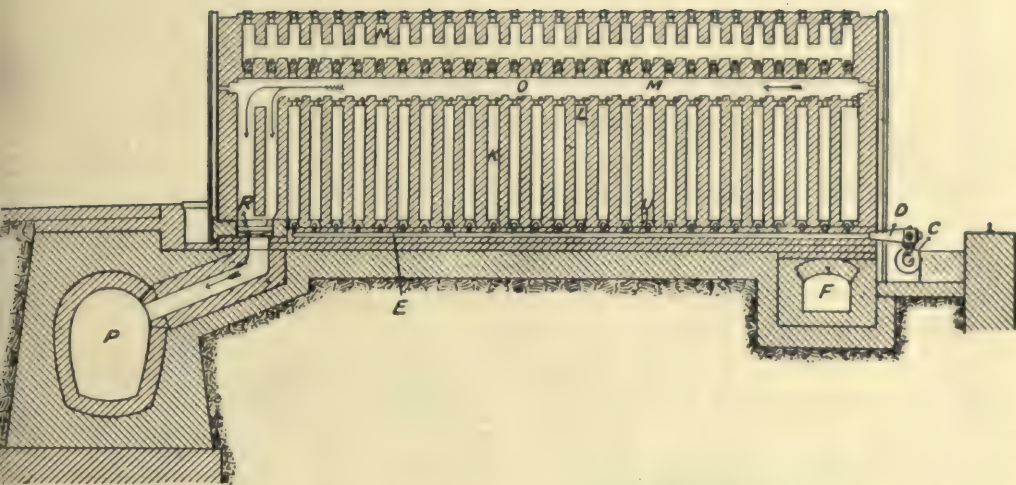


FIG. 49.—KOPPERS WASTE HEAT COKE OVEN: LONGITUDINAL SECTION THROUGH HEATING FLUE.

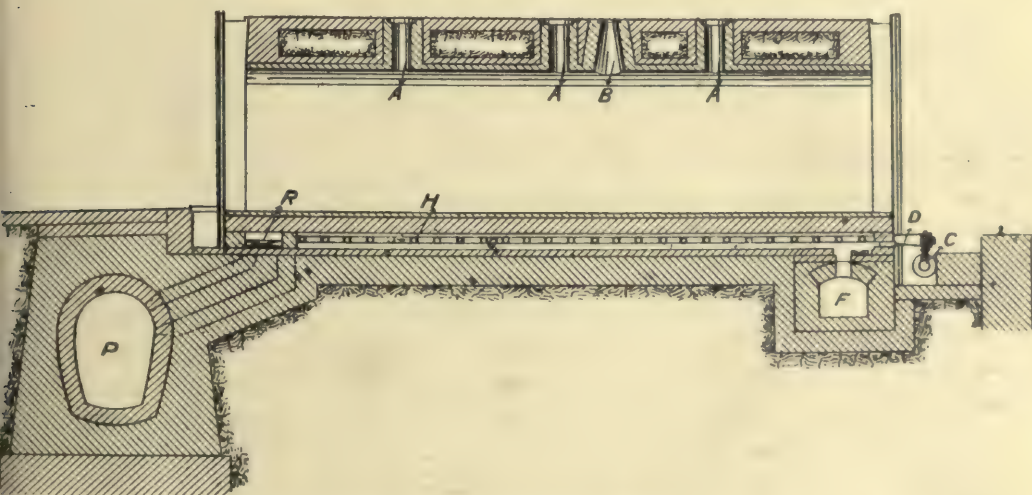


FIG. 50.—KOPPERS WASTE HEAT COKE OVEN: LONGITUDINAL SECTION THROUGH THE OVEN CHAMBER.

K, and through the openings, L, at the top of each flue. These openings are furnished with a damper, M, which can be regulated so as to enable the exact amount of air to enter the flue necessary to effect perfect combustion. The sliding bricks are accessible from the top of the ovens through the openings, N, which are fitted with easily removable plugs (Fig. 51).

The sliding bricks, and the openings at the top of the ovens which give access to them, are two of the principal features of the Koppers ovens. The openings at the top of the ovens serve not only to provide means for regulating the dampers, but serve more particularly to give access to the gas nozzles, U, and they further permit of the flues being inspected at any time. Without the facility thus provided for examining the flues, it would not be possible to work the ovens satisfactorily. The gas nozzles are furnished with oval orifices, to enable them to be taken out by a rod having a tee end (Fig. 53). The orifices in the nozzles vary in size, according to their position in the flues.

The Koppers Regenerative Coke-Oven.—The Koppers regenerative coke-oven is shown in Figs. 55 and 56. A careful description of this type of oven is contained in a paper by Mr. G. Stanley Cooper, B.Sc., before the Iron and Steel Institute, from which the following particulars are drawn.

The Koppers regenerative coke-oven possesses many special features, more particularly as regards the arrangements of the heating flues and regenerators, and the facilities for inspection. Figs. 55 and 56 show sections through the oven chamber and the heating flues. The length and breadth of the oven chamber are the same as in the waste heat type, the height and width varying somewhat according to the class of coal to be coked. Each oven is capable of taking a charge of about 8 to 10 tons of coal. The ovens are heated by the gas evolved from the coal during carbonisation, and the means adopted for distributing and regulating the supply of gas, together with the method adopted for pre-heating the air required for combustion, are special features. The gas and air for combustion are separately distributed on each side, and along the whole length of the oven, and combustion takes place in each vertical flue. Each oven-wall is formed of about thirty vertical flues, and there being a gas-jet in each flue, each oven is heated at about 60 points. At the top of the oven, and over all the vertical flues, openings are provided for inspection and regulation. Very simple means are provided for the regulation of the combustion in each flue, which is such that it is easily possible for the walls of the oven to be absolutely uniformly heated from end to end. This uniformity not only

Fig. 51.

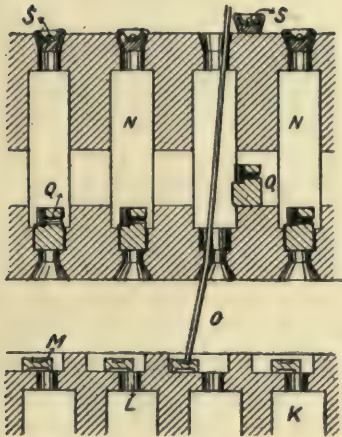


Fig. 53.

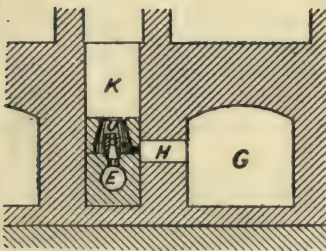
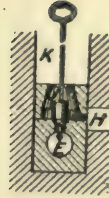


Fig. 52.

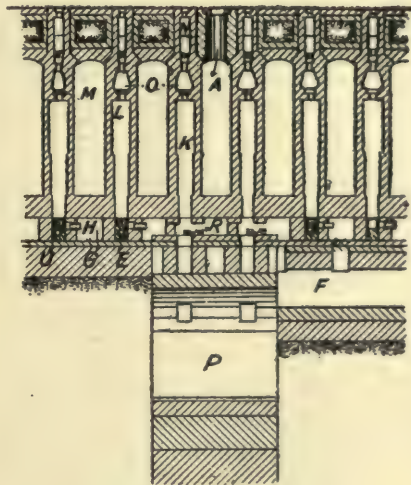


Fig. 54.

KOPPERS WASTE HEAT COKE OVEN.

FIG. 51.—Longitudinal Section through Heating Flues.

FIG. 52.—Cross Section through Gas Nozzle.

FIG. 53.—Cross Section through Gas Nozzle.

FIG. 54.—Three Cross Sections through Ovens.

results in the production of homogeneous coke, but enables the charges of coal to be burnt off more quickly than is the case when such uniformity does not prevail; besides which it effects great economy in the consumption of the heating gas. The air for combustion is heated to a high degree in regenerators designed on Siemens' principle.

The distinguishing feature of the Koppers regenerative oven lies in the employment of separate regenerative chambers for each oven, so that every oven is entirely independent of its neighbour. This construction permits of the air for combustion and the chimney draught being separately regulated. In the older type of regenerative oven there is a regulating damper over the passage leading from the regenerator chambers to the air-distributing channel. This damper has to be set to serve the dual purpose of regulating the admission of air when the ovens are burning in one direction, and of regulating the chimney draught when the ovens are burning in the reverse direction. It is not easy to effect a satisfactory regulation of both the air and the draught by means of one damper only.

Referring again to Figs. 55 and 56, the air for combustion flows along the passage-ways A, at the front and back of the oven, and passes thence into the regenerators through the inlets. In the regenerators the temperature of the air rises to 1000°C . (1832°F .). The highly heated air passes out of the regenerators into the vertical heating flues through the openings C. Part of the gas evolved from the coal is used for heating the ovens, and, after being purified, this gas is returned from the by-product plant to the ovens by mains D running along the whole length of the ovens on each side. Branch supply pipes H conduct the gas into the gas-distributing channels E, which are situated directly beneath the oven walls; thence it passes through the gas nozzles F into each vertical flue, where it ignites with the hot air entering through the passages C previously referred to. A jet is therefore formed on a level with the oven floor in each of the heating flues of the oven chamber. The gas is supplied to each oven by the branch-pipes, which are each provided with a regulating cock, as well as with a second cock fitted with a lever. All the levers are attached to one of the wire ropes, which communicate with the automatic changing gear. The air is admitted to each oven by means of the cast-iron damper fitting, which also serves to conduct the waste gases to the chimney flue. These dampers are each fitted at the top with a slide, connected, like the levers of the gas-cocks, with the second wire rope; and the whole arrangement is such that, when the automatic changing gear operates, the gas-cocks and damper-slides on one side of the battery are closed simultaneously with the opening of the correspond-

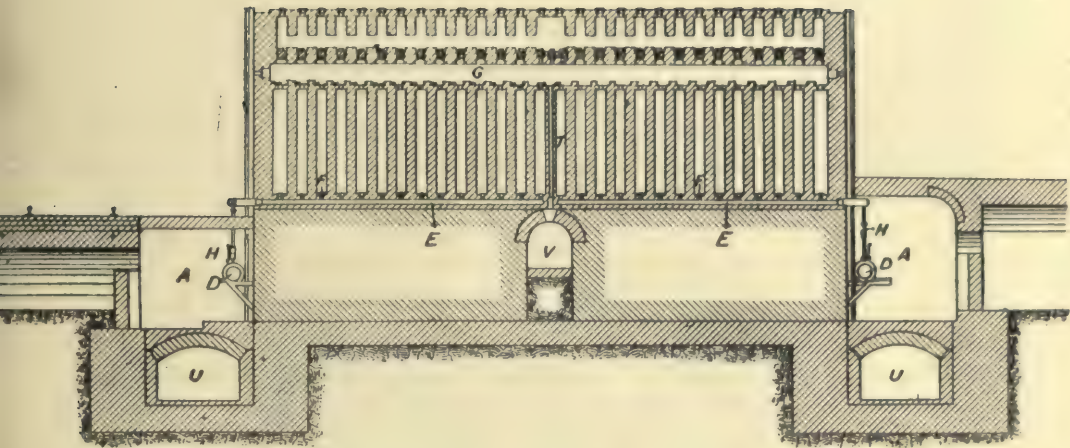


FIG. 55.—KOPPERS REGENERATOR COKE OVEN; LONGITUDINAL SECTION THROUGH HEATING FLUES.

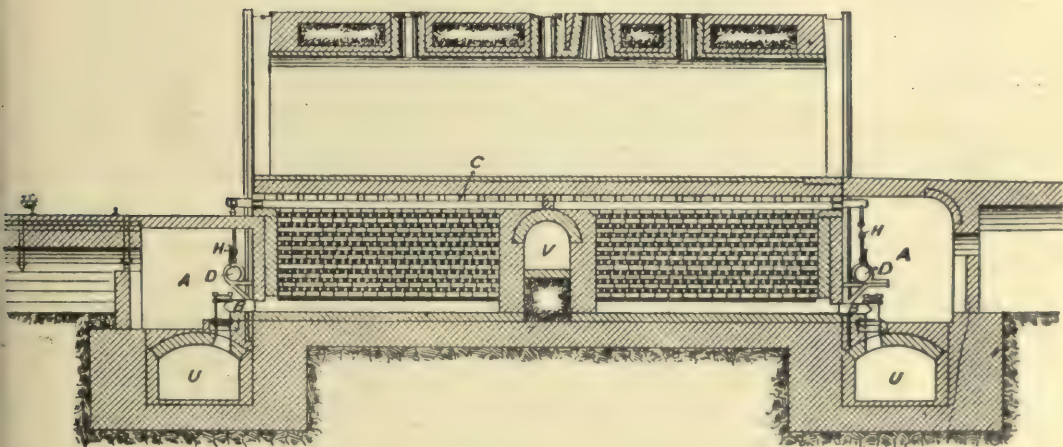


FIG. 56.—KOPPERS REGENERATOR COKE OVEN; LONGITUDINAL SECTION THROUGH THE OVEN CHAMBER.

ing parts on the other side of the battery. When the gas is burning on one side of the ovens the air-damper is open, and the air passes into the regenerator, the main chimney damper being closed. A little below the damper-slide there is a second slide, which can be set in any desired position, and serves to regulate the quantity of air required for each oven. When the gas is burning on the opposite side of the oven—that is, when the waste gases are passing to the chimney—the gas-cocks and air-dampers are closed, but the main chimney damper is open. The waste gases are therefore drawn through the cast-iron damper fitted into the chimney-flue. At the base end of the cast-iron fittings there is a third slide, which can be set in any desired position to regulate the draught on each oven. It will therefore be seen that by this arrangement the quantity of air for each oven, and the chimney draught on each oven, can be separately regulated, which is not possible with ovens working with the older form of regenerators. A very disadvantageous feature has thus been removed in the new system.

The automatic changing gear is a simple arrangement, and it is placed at one end of the battery of ovens. It is operated electrically or by hand, and requires the least possible attention. It may be added that the gear is so designed that should anything fail to act an electric alarm is set in action, and does not stop until the gear is again at work.

The employment of regenerators renders it necessary to reverse the heating process after a period of time, usually about thirty minutes, and the heating flues are divided into two sections, so that combustion can take place alternately in each half of the oven wall. When the gas is burning in one half of the wall, the products of combustion pass up the flues and enter the top horizontal flue G, whence they make their way down the flues in the other half of the oven wall, and enter the regenerator through the same passage C, by which the air is admitted to the flues when the direction of combustion is reversed. On issuing from the regenerator the waste gases pass into the flue leading to the chimney, after having given up their heat to the firebrick chequer-work. When the next half-hourly reversal is made, the heat is given up to the air entering for combustion.

The Koppers Horizontal Chamber Oven.—During recent years much attention has been devoted to various systems of carbonization in horizontal, inclined, and vertical retorts, and also in large chambers. The great improvements which have been effected are the result of endeavours to render the working of the plants as economical as possible by the introduction of mechanical labour-saving arrangements, and by the perfection of the methods of heating the charges. With the introduction

of incandescent lighting the object of the gas engineer has ceased to be the production of a low yield of rich gas of 20 candle power, and instead the aim is a high yield of, say, 14-16 candle power gas of good calorific value. The ideal of the gas engineer is therefore a system of carbonization which, while it possesses all the mechanical advantages which tend to labour-saving, must yet be such a system as can produce a large yield of good gas of even quality and suitable calorific value. At the same time it is further required that the yield of tar and ammonia shall be as high as is consistent with economical working, and that the setting shall be able

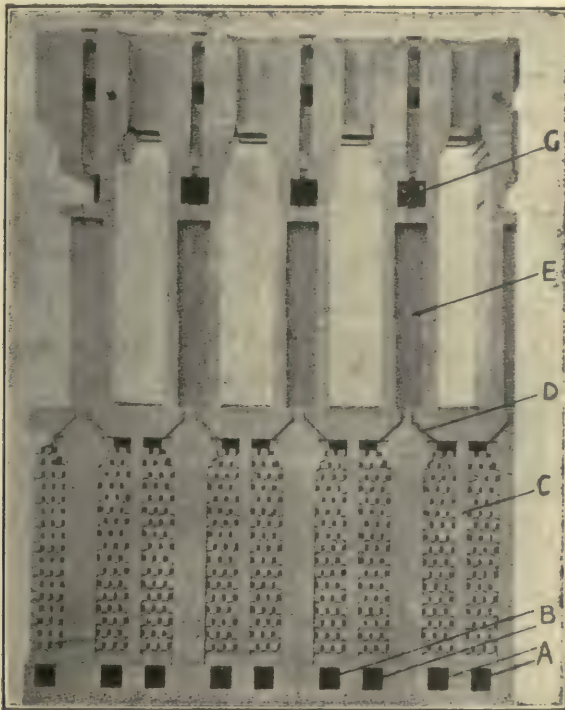


FIG. 57.—KOPPERS CHAMBER OVEN: CROSS SECTION.

to withstand satisfactorily the high heating required for the production of a large yield of gas.

The cost of working any particular system is largely influenced by the fuel costs. The percentage of fuel required for the satisfactory carbonization of the charges must be as low as possible in order to keep down working costs, and at the same time the heating must be even and well under control, so as to lengthen the life of the setting. The quality of the coke which is produced is also an important factor, and the minimum amount of breeze and the maximum amount of saleable coke are required.

It is claimed for the Koppers horizontal chamber oven that it fulfils all these requirements. At the Birmingham Gas Works 66 ovens have been installed, with a production of nearly 8 million cubic feet of gas per day.

Fig. 57 shows the horizontal chamber oven, which is a develop-

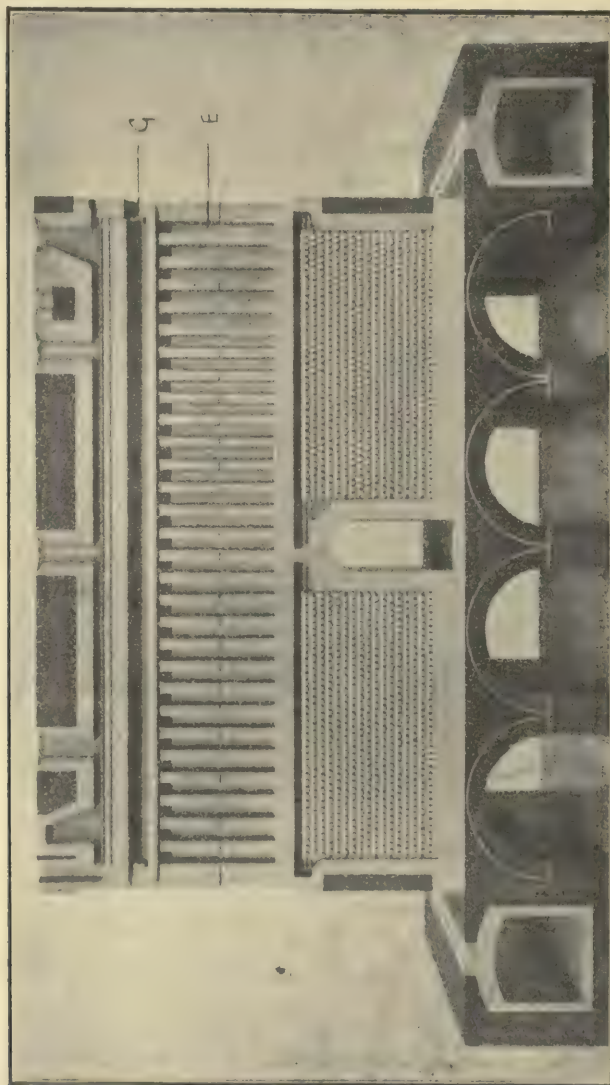


FIG. 58.—KOPPERS CHAMBER OVEN : LONGITUDINAL SECTION THROUGH HEATING FLUES AND REGENERATOR.

ment of the regenerative coke oven already described. By slight alterations in design and practice, the regenerative coke oven has been rendered capable of producing a high yield of gas of good and even quality, and suitable in every way for "town" purposes, while the coke obtained is

claimed to be superior to ordinary retort coke. The most important alteration necessitated is that the whole of the gas evolved from the charge shall be available for illuminating purposes, and hence some other means must be found for heating the ovens. In the Koppers chamber oven this is done by the introduction of a central producer gas plant erected quite apart from the chamber ovens. The producer gas has further to be pre-heated before combustion, just as the air is pre-heated in the case of the regenerator coke oven. Consequently the design of the oven is altered slightly in order to accommodate these new features. In the main, however, especially as regards the arrangement of the heating flues, the oven remains unaltered.

Fig. 58 shows a cross section through the heating flues and regenerator. In the gas oven the regenerator chambers are arranged so that gas is pre-heated in one regenerator, air in the next, then gas, and so on throughout the length of the battery. Each regenerator is further divided into two sections by a partition wall C, each section being connected by a pipe D to the heating flue E. Air enters through the pipes AA, as in the case of the coke oven, and producer gas through the pipes BB. The gas and air, after passing through the chequer brickwork in the regenerator, and becoming heated to about $1,100^{\circ}\text{C}$., issue from their respective pipes into the heating flue E, where combustion takes place. As in the case of the coke oven, combustion takes place alternately in each half of the oven wall, the products of combustion pass into the horizontal flue G (Fig. 58) down the vertical flues of the second half of the oven wall; hence passing into the regenerator chamber. After giving up the surplus heat to the brickwork of the regenerator the gases pass away to the chimney at a temperature of about 235°C . Owing to the fact that the producer gas as well as the air for combustion is pre-heated, great economy in consumption of the heating gas is effected, thereby reducing the fuel charges very considerably.

In connection with the whole of the installations of the Koppers chamber ovens on the Continent, the Kerpely gas producer is employed. This producer is described in the next chapter. The advantages claimed for the Koppers system of carbonisation in horizontal chamber ovens are (1) considerably lower labour costs owing to the use of mechanical appliances, and to the smaller number of charging and discharging operations; (2) low fuel consumption and cost, as the ovens can be heated by gas produced from coke breeze and low grade fuel; (3) the coke can be used for metallurgical purposes; (4) the yield of ammonia is considerably increased; and (5) the tar produced is of a light fluid character, and contains a very small amount of free carbon.

The Koppers Combination Coke and Gas Oven.—This type of oven is designed to meet the requirements of steel works desiring to use coke oven gas as fuel. Blast furnace or producer gas is not entirely satisfactory for use in steel furnaces; on the other hand, the value of coke oven gas has been demonstrated, the relatively small proportion of nitrogen and the high calorific value resulting in higher efficiency. With the ordinary type of coke oven, however, sufficient surplus gas is not produced to meet the full requirements of the furnaces, as from 40% to 50% of the gas evolved from the coal is required for heating the ovens, the balance only being available for outside purposes.

In describing the design and working of the combination oven, reference must be made to what has already been said of the regenerative coke-oven and the regenerative gas-oven. Plate VIII. shows cross-sections through (A) the Koppers regenerative coke-oven, (B) the Koppers gas-oven, and (C) the Koppers combination oven.

Coke-Oven A.—In this type of oven a portion of the gas evolved from the coal is used for heating the ovens, about 40 to 50 per cent. of the total quantity evolved being required. After passing through the by-product plant, the required portion of the gas is returned to the ovens through a series of distribution pipes G, through nozzles N, into the vertical heating flues F. Here it meets air which has been preheated to a high degree in the regenerators R, and combustion takes place. In this way the charge of coal is carbonised, and all the gas not required for heating purposes is available for outside consumption.

Gas-Oven B.—The Koppers gas-oven was designed for the production of the maximum quantity of illuminating gas, and hence none of the gas evolved from the coal is used for heating purposes. The ovens are heated by producer-gas generated in a central producer plant, and usually manufactured from inferior fuel. In this type of oven both the producer-gas and the air necessary for its combustion are preheated. For this purpose the generators are divided into two sections. The producer-gas is preheated by passing through the regenerators G R, and the air by passing through regenerators A R. The gas and air then meet in the vertical flues F, where combustion takes place as in the coke-oven.

Combination Oven C.—This oven is a combination of the coke-oven and the gas-oven, and can be used as either at will. The difference lies in the manner in which the oven is heated. If required as a coke-oven, it is heated by coke-oven gas; whilst if required as a gas-oven, it is heated by producer or blast-furnace gas. In the latter case the gas must be subjected to a cleaning process to free it from

tar or dust. It will be seen from the drawing that the combination oven
like to those adopted in the

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The Koppers Combination Coke and Gas Oven.—This type of oven is designed to meet the requirements of a coke oven.

tar or dust. It will be seen from the drawing that the combination oven is provided with divided regenerators similar to those adopted in the gas-oven. At the same time provision is made by means of the distribution channel G for the utilisation of coke-oven gas for heating as in the coke-oven. If the oven is to be worked as a coke-oven, all the regenerators are used to preheat the air. The coke-oven gas, freed from its by-products, enters the distributing flue G, and the oven works in exactly the same manner as described for the working of the coke-oven. If the oven is to be worked as a gas-oven, one pair of regenerators are used for pre-heating the air, and the next pair for pre-heating the producer or blast-furnace gas. The heated gas and air meet in the vertical flues E, and combustion takes place, the oven now working in exactly the same manner as described for the working of the coke-oven. The temperature of the oven can be maintained just as easily in one case as in the other, and the change from a gas-oven to a coke-oven, and *vice versa*, can be made almost instantaneously, and without interfering with its working in any way.

The Semet-Solvay By-Product Recovery Coke Oven.—The Semet-Solvay coke oven, as built to-day, is the best representative of the class having horizontal, instead of vertical, heating flues.

The Semet-Solvay oven is made in three distinct types, viz.:—
(1) That with the heating flues built into the solid supporting walls, as shown in Fig. 59. It retains the essential feature which distinguishes the Semet-Solvay from other ovens—namely, each oven has separate sets of flues and is separated from its neighbours by solid walls. With moderately dry coking coals containing over 27% of volatile matter the surplus gas is usually 30% of the whole. (2) That with the supporting walls independent (Fig. 60). The heating flues and roof form a lining to the permanent oven structure; in fact, the flues can be removed and rebuilt without affecting the main structure. This type is more expensive, but is preferred in many instances because of its substantial design and long life. Batteries are still at work which were built twenty-five to twenty-eight years ago. (3) That similar in construction to the first described, but with provision for pre-heating the air to a greater degree, as shown in Fig. 61. The air passes through a continuous regenerator, and the yield of surplus gas is increased up to 60%.

The oven chamber as now built is 8 ft. to 10 ft. high by 33 ft. to 36 ft. long, and varies in width from 16 in. to 22 in., according to the nature of the coal to be carbonized. On each side of the chamber, and

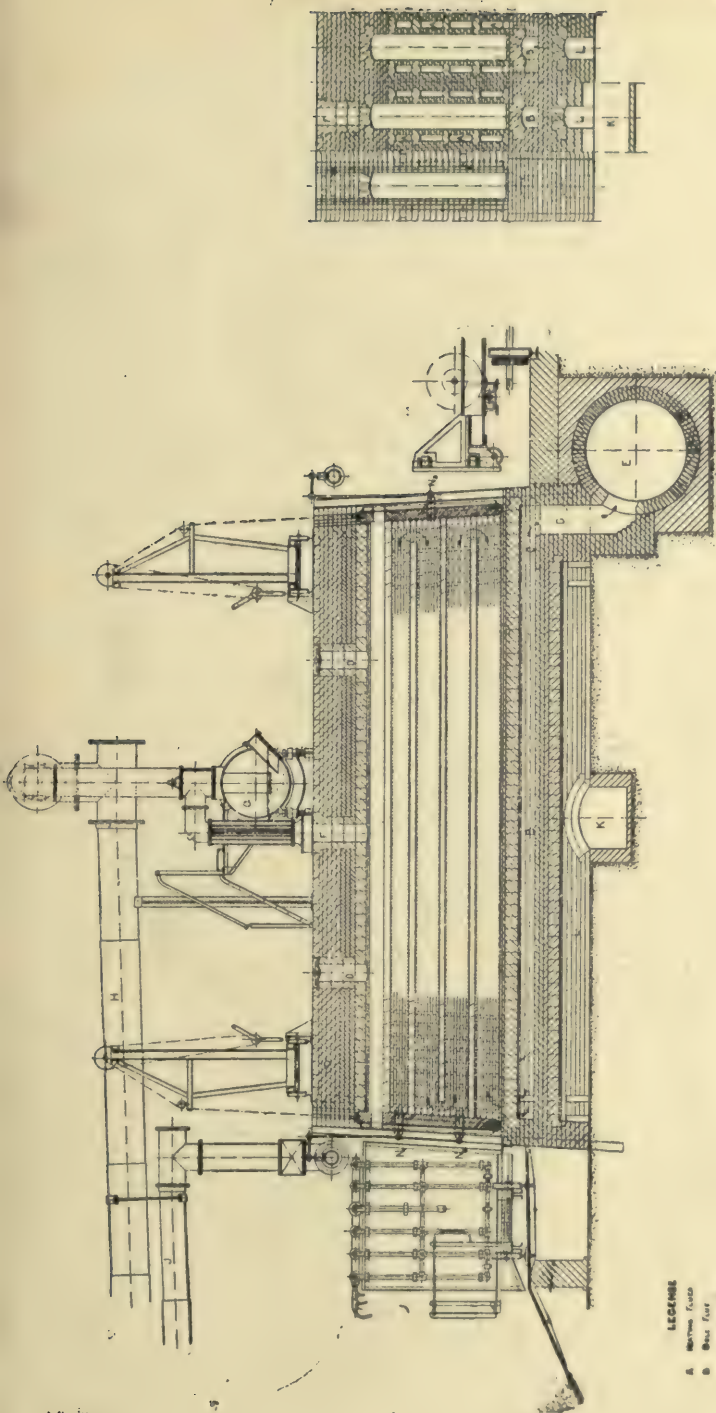
separated from it by walls 4 in. thick, are longitudinal heating flues A. Underneath the chamber is a sole flue B, in which the burnt gases from the flues on either side are united, to travel under the bottom of the oven, past the regulating damper C, and port D, into the waste gas flue E, in which they are carried to the boilers or chimney.

The gases evolved from the coal in the process of carbonization pass through the outlet F in the roof of the oven into the hydraulic main G, where, under the influence of an exhaustor, they bubble through weak tar and ammoniacal liquor, and are drawn through the green gas main H to be condensed and washed in suitable apparatus. The gases, having been thus deprived of the ammonia, tar, and light oils with which they were charged, are returned by the exhaustor through the scrubbed gas mains J to supply the fuel required to heat the oven walls. There is, however, more gas available than is necessary for this, and the surplus may be used for other purposes.

In the waste heat type of oven, Figs. 59 and 60, the air required to burn the gas in the flues is pre-heated, being drawn by the chimney draught under the whole length of the battery of ovens in flue K, and under each oven in the flues L, and delivered at a temperature of about 300° C. through the upcast ports M into the heating flues A. Gas is admitted into the heating flues, together with a regulated volume of pre-heated air, first at the point N₁, and successive reinforcements of gas and hot air are admitted at N₂ and N₃. The oven illustrated is designed for compressed charges of coal, served through the oven door, and the holes O, or M, in the crown of the oven, are not charging holes, but are intended to let off the gas emitted by the coal in the first few minutes of charging.

The oven shown in Fig. 61 is a departure from the usual principle upon which the regenerative type is constructed. It is a development of the waste-heat Semet-Solvay oven. A number of installations have been at work for some time in this country, on the Continent, and in America, and the makers state that strikingly good results have been obtained, and they give the following reasons to show that such results are only to be expected from the new departure.

Referring to the drawings, admissions of gas and hot air are made in the top flue at L₁, and in the second flue at L₂, and of gas only in the third flue at L₃. The burnt products travel through the fourth flue without further admissions, and unite in the sole flue B with the burnt products from the opposite wall of the oven, passing out at D to the main flue E through a continuous recuperator. The burnt gases travel along horizontal channels in the recuperator, and the air passes through vertical holes, absorbing the heat in its passage. The temperature of

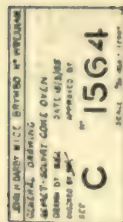


LEGEND

- A. Bottom Flues
- B. Blast Flue
- C. Breasting Down
- D. Waste Gas Port
- E. Main Waste Gas Flue
- F. Gas Outlet Hole
- G. Watercock Main
- H. Gases Cold Road
- I. Steam Cold Road
- J. Steam Hot Flue
- K. Main Air Flue
- L. Air Flue
- M. Main Air Flue
- N. Cold Burners
- O. INLET HEAT IN REAR



FIG. 59.—SEMET-SOLVAY BY-PRODUCT RECOVERY COKE OVEN: WASTE HEAT TYPE.



the burnt products leaving the recuperator and entering the main flue is about 300°C ., and that of the heated air at the burners about 550°C . Each oven has its own separate recuperator.

Unlike other regenerative coke ovens, this is not of the reversing type. The air is delivered continuously to the burners at a constant temperature, and there are no alterations of temperatures to affect the life of the brickwork in the heating flues or regenerators.

In the Semet-Solvay oven, gas and air admissions are made successively in the top and second flues, and gas only is admitted to the third flue. If the air be pre-heated to 550°C ., the theoretical quantity for combustion produces a temperature far in excess of the temperature which coke oven bricks are capable of resisting. It is therefore impossible to burn coke oven gas with the theoretical quantity of air, and consequently an excess of air is necessary in each of the top flues. This excess of air in the top flue renders its temperature lower than any of the others. But in traversing the flue its initial temperature is raised from 550°C . to about $1,000^{\circ}\text{C}$., at which temperature it passes along with the burnt products into the second flue. In other words, the air is, to that extent, regenerated *in situ*. This is as it should be, as it is generally accepted that a low temperature at the top of the oven favours the recovery of by-products. In the second flue, as the temperature of the burnt products derived from the top flue has to be raised to the temperature of combustion, the temperature may be regulated with very little additional air. In the third flue again, the burnt products have to be raised to the temperature of combustion, and no further admission of air is necessary, the gas being burnt with the free oxygen in the burnt products from the flues above.

To sum up, the total of the volumes of air delivered to the two upper flues is practically the theoretical quantity required for the total gas delivered to all three burners. It is claimed that in working the Semet-Solvay oven a minimum of burnt products is produced, and that of the total gas given off by the coal the quantity available as surplus is appreciably greater than is obtained from vertical flue ovens with reversing regenerators.

In each type of Semet-Solvay oven described it will be observed that each oven has its own proper heating flues, divided from those of the adjacent oven by solid thick walls; by this means the weight of the top brickwork and hydraulic main is supported independently of the red hot flue walls. If the maximum yield of coke and by-products is to be attained, there is no point in the construction of a coke oven of greater importance than the "gas-tightness" of the walls dividing the oven chamber from the heating flues. Owing to the change of tem-

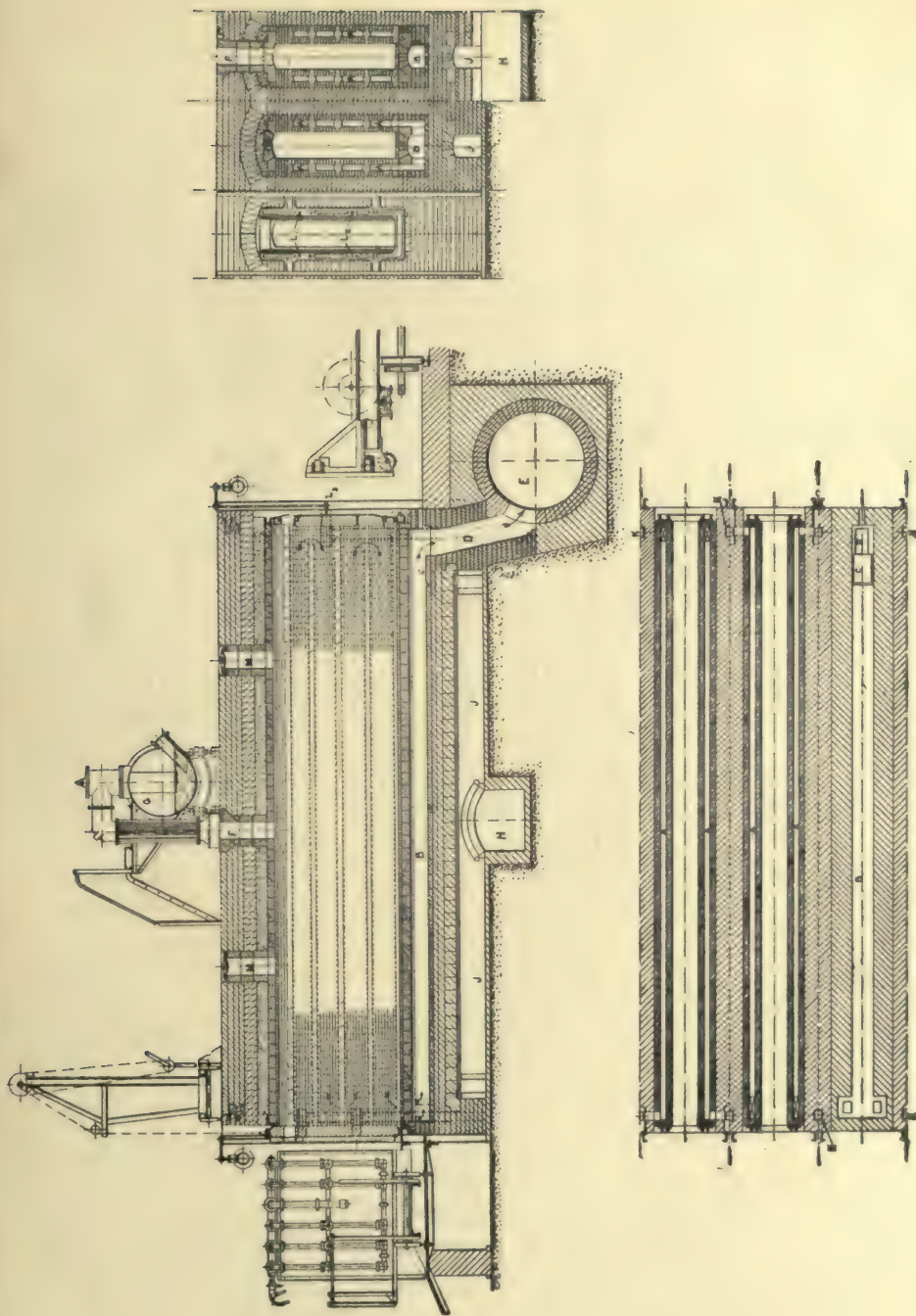


FIG. 60.—SEMET-SOLVAY BY-PRODUCT RECOVERY COKE OVEN: WASTE HEAT TYPE.

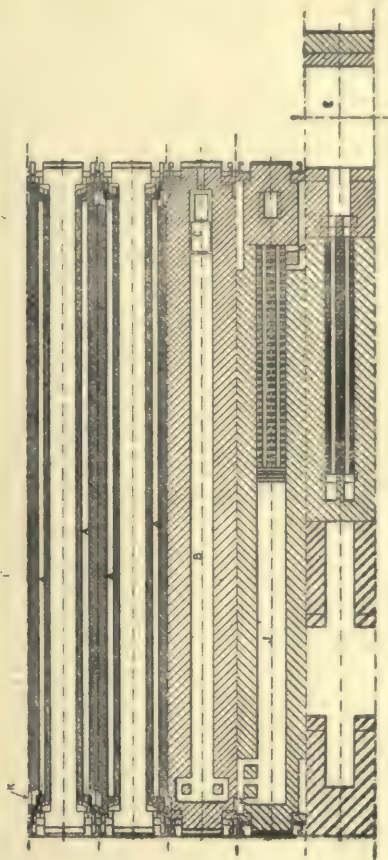
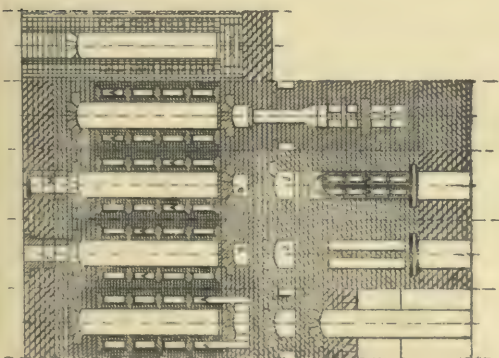
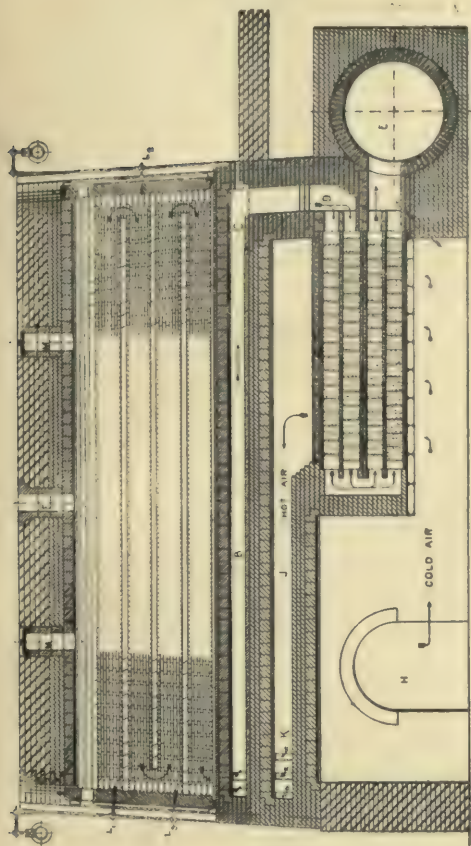
perature resulting from the introduction of every fresh charge the demand upon the inner linings is very severe. If the bricks used in the linings are too large, the movement of expansion and contraction to which they are subject brings about fractures and open joints; if sufficiently small, that movement is harmless in the individual brick. The heating flues in the Semet-Solvay oven are therefore built up of pieces measuring only 8 inches by 4 inches by 2 inches, a size which admits of their being manufactured with true plane surfaces. Such bricks are capable of being laid in place with joints of about $\frac{1}{8}$ -inch in thickness, and if they are not well enough made for such exact laying they are discarded.

A sight-hole is provided at each end of every heating flue, through which the gas flame, the degree of heat, and the condition of the walls are kept under continual observation.

The structural value of the solid dividing walls has already been mentioned, but they play an equally important part in the process of coking. They form a heat store, not directly affected in temperature when a fresh charge of coal is introduced into the oven. The thin flue walls next to the coal are naturally chilled by such a charge, and if they depended solely upon the heat of the gas burnt in the flues, their recovery of temperature would be slow; but they draw also upon the heat stored up in the dividing walls, and the oven is carried, as it were, over the dead centre very quickly. It is generally conceded that the Semet-Solvay oven is a quick oven, and it gains this reputation in its power of recovery from the chilling effect of a fresh charge. The dividing wall is, in fact, a recuperator. The advantage of this feature is particularly observed in the successful working of compressed charges.

In the Semet-Solvay system each oven is heated independently. The heating flues are horizontal, and the gas and air admissions are on the oven faces. Effective and equal distribution of the heat in each separate flue is in this way under observation and control. The gas enters in the top flue, and travels downwards into the sole-flue, licking the walls with a long flame, the speed of which is comparatively slow, since its course is downwards. An exact parallel exists in the nature of the flame in a reverberatory furnace. The chimney draught in this, as in most other types of ovens, is 8 to 10 m/m of water-gauge.

It has been stated that the gas admission end of horizontal flues is hotter than the opposite end. It is pointed out by the makers that if there were any truth in this statement the Semet-Solvay oven would have been condemned long ago instead of having been accepted with the favour it has received. The temperatures have been frequently recorded, and prove the statement to be incorrect. It has already been



| | |
|--------------------------------|------|
| THE COKI COKE DISTILLATION CO. | |
| DESIGNED BY | DATE |
| DRAWN BY | DATE |
| CHECKED BY | DATE |
| APPROVED BY | DATE |
| C 2664 | |
| SCALE 3/4" = 1'-0" | |

FIG. 61.—SEMET-SOLVAY BY-PRODUCT RECOVERY COKE OVENS; REGENERATIVE TYPE.

explained that so much air is admitted in the top flue that the excess is self-regenerated in its course along the flue, the products carried forward giving an equable temperature throughout its length. The temperature in the top flue is about 950°C ., in the second $1,050^{\circ}\text{C}$., and in the third $1,100^{\circ}\text{C}$. It has been demonstrated by long experience that the cost of keeping the Semet-Solvay oven in repair is extremely low. It is substantial in design, and its construction makes it easy to repair.

In some places the practice is still followed of filling the ovens through holes in the roof, and levelling the charge by hand or power. To do this it is necessary to have one or two more charging holes than are shown in the illustrations given of the oven. Where charging plant is used, the charging machine travels upon the same track as the coke ram, and may be described as a box, the dimensions of which are a little smaller than those of the oven. Its bottom is formed of the charging peel, having a flat upper surface and rack teeth underneath, and a thrust plate at its back end of the same height as the box. The front end of the box is closed by a hinged door until the operation of stamping or compressing is completed. The stamping machine runs on a superstructure mounted on the charging machine, or alternatively on a girder thrown at a convenient point across the ram race, under which the charging machine may take up its position. While the loose coal is being served into the box the stamping machine moves backwards and forwards along the length of the box, reversing itself automatically at each end and compressing the coal, until the box is full. The oven door being opened, the sides of the box are slightly released by suitable mechanism, and the charging peel, being put in operation, carries the stamped cake of coal into the oven. The thrustplate at the back of the peel is then removed, the oven door closed, and the peel withdrawn, leaving the coal in the oven.

The charging machine may be combined with a coke ram. The combined machine is operated by one man, and is advisable where the number of ovens is not great. But for the rapid and effective service of a large number of ovens it is questionable whether it is not better to separate the machines, as in the case of the combined machines delays in the working or repairs to the parts of one side suspend the use of the other for the time being.

The arrangement of the compressing plant is subject to many variations, dependent principally on the position of the coal storage bunker in relation to the ovens. When the number of ovens is considerable, a rubber band conveyor is used to supply the compression box, carrying up to 100 tons of coal per hour. The coal is taken off the

conveyor by an automatic reversing distributor ranging over the length of the compression box. The chargers are designed to pass to and from underneath the stamp and conveyor run-way which spans the ram race.

Two independent stamping machines are preferable to the ordinary double stamp. The latter reduces the time taken by a single stamp to compress a charge, but it by no means halves that time, as separate machines do. Independent machines reverse automatically on meeting each other, the meeting place varying with each return journey, and in case one is temporarily broken down the plant is still kept going by the other.

The service bunkers now supplied with most Semet-Solvay plants are of tank form, hoppers at 60° to one outlet, where the coal is drawn by a revolving feed-table.

When the process of coking is completed there remains in the coke about 0.5% of volatile matter. The time each charge takes varies from twenty-two to thirty hours, according to the class of coal and width of oven. The batch of coke is then pushed out by a coke ram. The ram returns out of the oven five times faster than it enters, the reversal being made, not by the motor, but by means of friction clutches.

The batch of coke in passing out of the oven goes through a Darby patent quencher with a large number of water jets. These jets quench the coke before oxidation in the air can take place. It is this oxidation which gives by-product coke its dark appearance. Quenched thus, the coke has the same bright and silvery appearance as "beehive" coke, and contains under 1% of water. The quencher may be mounted on a car running on a track along the discharge hearth, or, alternatively, suspended from an overhead rail.

Emerging from the quencher the coke is received upon an inclined hearth. If it is to be loaded with forks direct into wagons, there is a short level platform or standage place at the foot of the slope. If, as the situation of the ovens determines, it is to be loaded into blast furnace barrows, there are gates at the foot of the slope for that purpose. Or if the coke is to be carried away by a collecting conveyor, the conveyor will take the place of the gates. Such slopes are preferably plated, as then the angle selected is that at which the coke will come sliding, and not tumbling down. If they are laid with brick setts the inclination at which the coke will slide is rather too steep, and the coke is broken up more. If the coke cannot be loaded into trucks on a siding in front of the bench, or if it is to be screened, it may be transported to a loading station either by a conveyor, or by the makers' patent coke-car.

The Otto Coke Oven.—The Otto waste heat by-product coke oven is shown in Fig. 62, and the Otto regenerative oven in Fig. 63. A description of the model plant of the Otto Coke Oven Co., as installed at their Crigglestone Collieries, will indicate clearly the main points of the Otto system.

The coal from the Crigglestone mine, owing to the thinness of the seams and the disturbed nature of the ground, cannot be worked for sale in the usual way, but the profits on by-product coking have made it possible to work the seams to approximately the full capacity of the mine—equivalent to 1,200 tons of coal per day. The large coal down to $1\frac{1}{2}$ in. is screened and cleaned on picking belts and sold for household purposes. The small coal below $1\frac{1}{2}$ in. is brought from both shafts by indiarubber belts over automatic weighing machines to the top of a storage bunker built in ferro-concrete, which has a capacity of 700 tons. From this bunker the colliery coal falls into the pit of the washery elevator, where it is mixed with coking slack from other collieries, brought in wagons and tipped by means of hydraulic plungers. In this way any desired mixture of coals of different coking quality is assured.

The washery is of the Luhrig type, 50 tons capacity per hour. The small coal is first sized into engine nuts, $1\frac{1}{2}$ in. to $\frac{7}{8}$ in.; beans, $\frac{7}{8}$ in. to $\frac{5}{8}$ in.; pea nuts, $\frac{5}{8}$ in. to $\frac{3}{8}$ in.; small, $\frac{3}{8}$ in. to 0 in. The smaller sizes are washed in nine felspar washing boxes, and then elevated into fourteen draining hoppers of 100 tons capacity each, where the moisture is reduced to approximately 12% by drainage. The washed nuts are either delivered into wagons outside the washery building, for sale, or they can be mixed with the coking small. The ash in the small coal is reduced by this washing process to 5% or 6%. The shales which are washed out contain not more than 2% of free coal. The drainage coking coal is brought by scraping conveyors and elevators to a Carr disintegrator of 35 tons capacity per hour, and then elevated to a crushed coal loading bunker of 300 tons capacity, which serves simultaneously for loading coal into the stamping box and also into tubs for filling the ovens from the top. In this way coking trials can be made both with compressed or uncompressed coal. The plant consists of fifty waste heat with five experimental ovens, and to these recently have been added twenty-five ovens on the regenerative principle.

The waste heat ovens (coking chambers 33 ft. long, 6 ft. 10 in. high, and 21 in. wide in the middle) are usually charged with stamped coal. Through thus compressing the coal, a cake of over nine tons can be charged into an oven, the coking time of which varies from thirty-four to thirty-six hours. The chambers of the new regenerative ovens

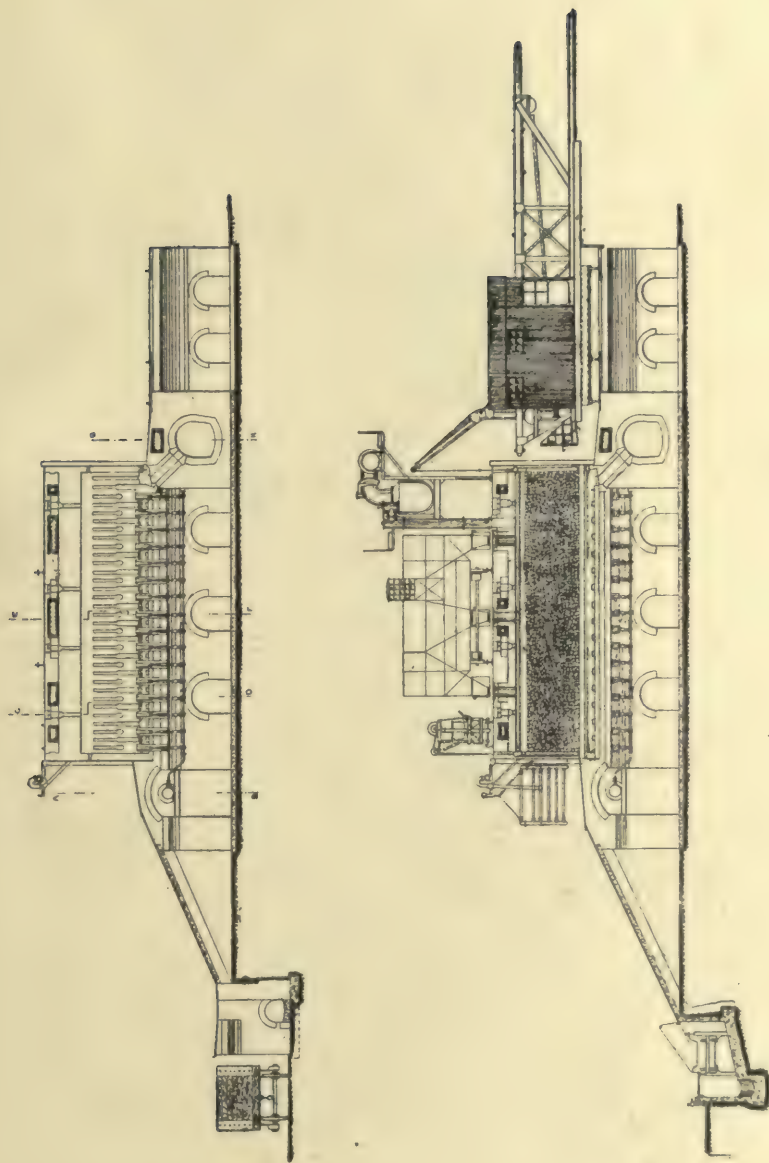


FIG. 62.—THE OTTO BY-PRODUCT RECOVERY COKE OVEN: WASTE HEAT TYPE.

are of different widths, in order to suit any class of coal, and are 8 in. higher than the waste heat ovens. These regenerative ovens are usually fed with coal from the top, through three charging holes, and levelled mechanically, but compressed charges can also be coked in them if desired. The coking time of the top-filled ovens is twenty-four or thirty hours respectively, according to the difference in widths of the coking chambers. The coke from both the waste heat and the regenerative battery is pushed through an open type quencher on to an inclined bench, and loaded into trucks mechanically over a coke conveyor and screening plant, but the two kinds of coke can be loaded separately in accordance with the requirements of customers. The yields of the mixture of coal treated are approximately 72% of coke, including breeze, 1.4% of sulphate of ammonia, 3.7% of tar, and 1.1% of benzol and homologues (toluol, xylol and solvent naphtha). The distillation gases are collected in a gas main on the top of the ovens, and from there are drawn through the by-product recovery plant, where first the gases are cooled, and the gas liquor and tar is condensed. After this the gas passes through a special spray and a final scrubber for the full extraction of the ammonia, and afterwards through three benzol scrubbers, where it meets with creosote oil for the extraction of the benzol products from the gases. The gas thus freed from impurities and valuable by-products, is returned into the cellar underneath the ovens, where it is burnt in the oven walls, thus providing the heat necessary for the distillation of the coal. Each wall of the waste heat ovens is heated by fifteen Bunsen burners, and the waste heat passes through two of the three Babcock and Wilcox boilers (150 lbs. pressure, 500° F. super-heat) of 4,750 square feet heating surface each, before passing up the chimney. The accessibility of each heating point in cellars underneath the ovens is the principal feature of the Otto ovens, and this is the same in the new regenerative ovens, where the waste heat passes through regenerators which alternately pre-heat the air, and thus save 50% to 60% of the gas which would otherwise be used for keeping up the temperature of the ovens. This surplus gas is used for firing four Lancashire boilers, 80 lbs. pressure, which form part of the old colliery equipment. The high pressure steam obtained from the waste heat is partly used for the requirements of the colliery, washery, and the by-product recovery plant, but mainly for the generating of electricity. Two sets of 500 K.W. Westinghouse generators, 500 volt, direct current, from the power station, together with a 150 K.W. generator as a stand-by. While the ovens, both waste heat and regenerative, are of the latest type, the by-product recovery plant in operation at Crigglestone

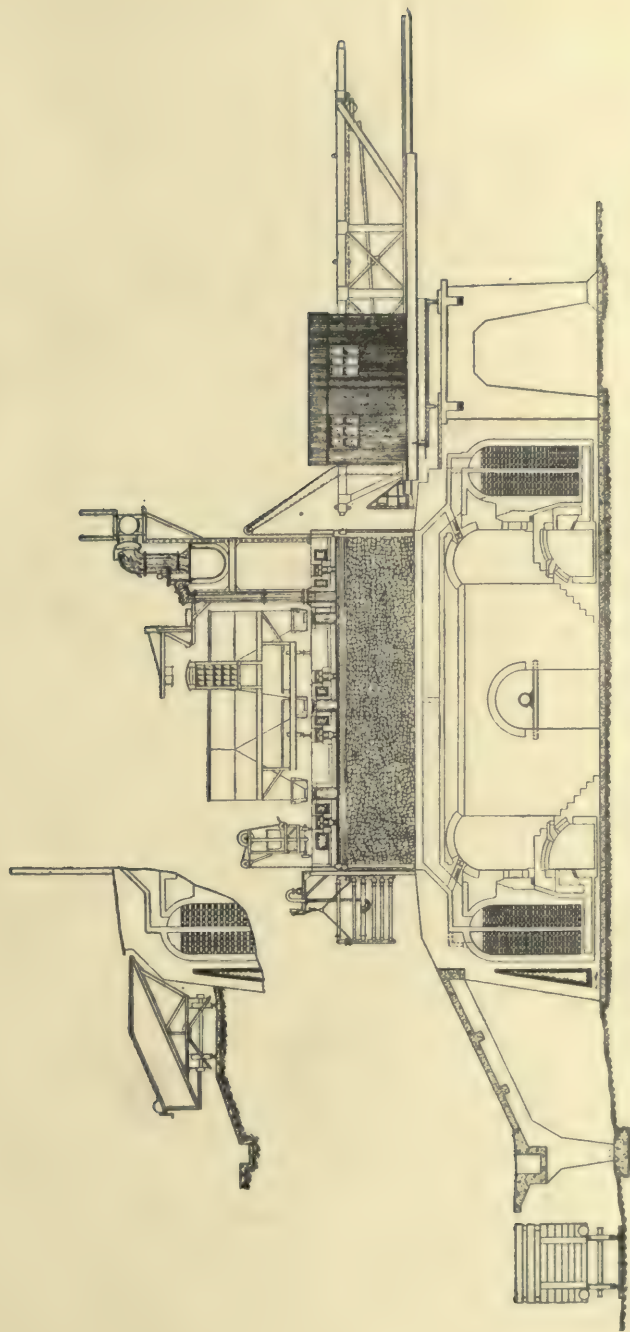


FIG. 63.—THE OTTO BY-PRODUCT RECOVERY COKE OVEN: REGENERATIVE TYPE.

has been superseded in recent years by the Otto direct recovery plant, which is described in Chapter I.

One of the most recent installations of the Otto system is that of the Team By-Product Coke Company at Dunston. This plant, which comprises washery and 120 Otto regenerative ovens, was fully described in the *Iron and Coal Trades Review* of May 7th, 1915, from which the following particulars are taken.

The coal brought in passes to the top of the full sidings, where is installed a Pooley weighbridge (50 tons capacity) from which the trucks gravitate to the point of discharge immediately below the washery. The containing building for the coal-handling and washing plant is a steel structure, lined with red brickwork; the whole of the machinery is erected upon steel girders, and the stairs, ladders, and flooring are steel chequered plates. The plant is very comprehensive, providing as it does for the storage of washed and unwashed coal—the capacity of the washer is 80 tons per hour—draining and disintegrating.

The coal brought to the washery building is discharged into one of two boots, one for the coal which analysis has determined does not require washing, and the other for the coal to be passed through the washery. Each of these pits is served by its elevator, the two elevators having a joint capacity of 140 tons per hour. The clean coal is elevated direct into the storage bunkers, and the coal for washing into a set of shaking screens, which are arranged to separate the small coal from the nuts. The small coal passes into the mechanical distributor, and thence to the washer, and the nut coal into a scraper conveyor discharging into the distributing conveyor over the drainage bunkers, where it is mixed with the washed coal. The washing plant consists of 48 of Messrs. Campbell, Binnie, Reid and Company's improved felspar washing tanks, which are arranged in eight nests of six, an interesting feature being the mechanical feeding apparatus for feeding the coal and water into the felspar tanks. By means of this simple but effective apparatus the makers are able to ensure that each tank receives an equal supply of coal and water, as also that the coal and water are thoroughly mixed before entering the tanks, both obviously important considerations.

After washing, the coal is discharged along with the water into a pair of the makers' special settling tanks of large capacity, and each fitted with a slowly travelling bucket elevator of the dredger type, perforated for drainage, and with a capacity of 40 tons per hour. The settling tanks are of a special design, the receiving and discharging arrangement and diaphragms being of a construction calculated to take full advantage of the total available settling area, with the result that

whilst the tank receives a large quantity of water heavily laden with coal, it passes the discharged water in a state comparatively free from suspended matter. The coal recovered from the water is discharged from the elevator into a conveyor delivering into the distributing conveyor over the drainage bunkers, where it meets and mixes with the nut coal from the shaking screens. The distributing conveyor, which has a capacity of 100 tons per hour, runs the whole length of the drainage bunkers, and is fitted with a series of hinged doors for discharging its contents into the various compartments of the drainage bunker. The conveyor is driven by a Holmes motor of 19 h.p. at 775 r.p.m.

The drainage bunkers, including those set apart for the unwashed coal, have a total holding capacity of 3,400 tons. They are divided into 46 compartments arranged in parallel lines of 23 each. They are built of brick and cement, the bottoms being formed of inclined arches terminating in swing discharge doors easily operated by hand.

Each line of hoppers delivers on to a scraper conveyor which discharges into a cross-conveyor for feeding into the disintegrating plant. Each of the conveyors has a carrying capacity of 40 tons per hour, and is driven by a Holmes-Clayton 19 h.p. motor running at 775 r.p.m. This arrangement of bunkers provides for feeding the coal in any desired quantity from each and every hopper, whilst a complete stand-by to this arrangement is furnished with the second line of hoppers and their conveyors. The disintegrators are housed in a walled-off building. The two sets (one a stand-by) have each a capacity of 50 tons per hour. They are of the Carr type, but of exceptionally heavy construction throughout. Each set of disintegrators is driven from belts by two motors, each of 65 h.p. The coal, after passing through the disintegrators, is elevated and discharged into the crushed-coal bunker, placed above and between the batteries of ovens. The elevator serving this bunker is driven by a 30 h.p. motor.

The 120 Otto regenerative ovens are arranged in two batteries, between which is placed the crushed-coal bunker and the coke-screening plant. Each battery of ovens has a stack 175 ft. high by 7 ft. internal diameter. The two batteries are served by three rams and levellers, an arrangement which provides a common stand-by. The machines are provided with electrically-driven jib cranes for raising and lowering the doors on this side of the ovens. Each oven is capable of taking a charge of $8\frac{1}{2}$ tons of washed coal, the time required for coking which is approximately 28 hours; the installation is therefore equal to coking 6,000 tons of washed coal per week. The ovens are approximately 33 ft. 7 in. long by 7 ft. 11 in. high, with a mean width of 1 ft. 9 in. As

is the case with all the Otto ovens, the gas is led to the heating flues from underneath the ovens by means of 15 connections from each oven wall, the gas being thus evenly distributed throughout the whole length of the walls, ensuring even heating. All the connections and the reversing gear are easily approachable, and any of the vertical flues can be inspected. The ovens are charged by electrically driven chargers, which are fed from the crushed coal bunker, and discharged into the ovens by gravity. These machines carry a full charge for one oven, and either machine can, if necessary, travel the whole length of both batteries of ovens. The coke is discharged through an open-type quencher, provided with a Cochrane anti coke breaker, on to a sloping bench; thence sliding to the coke conveyor by which the coke from each battery of ovens is delivered to the central screens. These conveyors, each 260 ft. long by 36 in. wide, are of Campbell, Binnie, Reid and Company's special tray type. They are provided with the Otto system of tilting plates hinged on the bench side, which are normally in the vertical position, so as to throw off any water draining down the bench when the conveyor is not actually receiving its load. Features of the conveyor design are the exceptionally large wearing surfaces and the facility for renewals.

The coke screens, of the shaker type, are 7 ft. wide, made in two sections driven by eccentrics arranged at opposite sides on the same shaft, so that one section of the screen balances the other, and vibration is almost eliminated. The screens separate the coke into three sizes—ballast, breeze and large. The two first-named are delivered into separate hoppers placed over the wagon roads, and the large coke is delivered on to a plate-type loading band, provided with a hinged lowering shute.

In the paper read before the Institution of Mining Engineers by Mr. Ernest Bury, already referred to in Chapter I., the use of coke-oven gas for town lighting is referred to, the application of the Otto direct-recovery process in that connection being discussed. Mr. Bury observed that it had been suggested that by the passage of the gas through hot sulphuric acid in the recovery of sulphate, the illuminating and calorific value of the gas would suffer through loss of the heavy hydrocarbons. Mr. Bury examined this point at Auchengeich, and found the calorific value of the gas on several days' tests to range as follows: 596, 602, 599, 608, and 581 British thermal units per cubic foot; these figures are comparable with those of present-day town gas. Mr. Bury had inspected the Hanover III. Otto plant, working on the direct-recovery process, which is supplying the town of Bochum with illuminating gas. The following are the details of this installation:—The

plant had been working since December 20th, 1910, with perfectly satisfactory results. Fifty Otto regenerative ovens were in operation, with a capacity of 5 tons of coke per oven per day. The illuminating gas plant was of a size to produce 70,630 cubic feet (2,000 cubic metres) per hour, and when the plant is extended to 100 ovens, it will be able to supply 141,260 cubic feet (4,000 cubic metres) per hour. The total volume of gas with the full capacity of the battery amounted to about 3,531,500 cubic feet (100,000 cubic metres) per 24 hours, so that with a yield of 1,695,120 cubic feet (48,000 cubic metres) of illuminating gas per 24 hours 48% of the total volume of gas could be given off as illuminating gas. The highest daily supply hitherto reached with a maximum heating value of 5,300 calories had been 1,341,970 cubic feet (38,000 cubic metres) per 24 hours, that is, 38% of the volume of the total gas. The ovens were jointed with korondin, this being the only precaution taken to ensure the gas tightness of the oven walls. In order to draw off the illuminating gas separately, each ascension pipe was provided with a second valve, connected with a special main. The separation of tar and ammonia was accomplished in the usual way by the direct-recovery process, the gas being drawn by means of a Connersville injector through the tar spray apparatus and the saturators, and afterwards through two coolers with a cooling surface of 3,225 square feet (300 square metres) each. The gases were cooled down to 48° or 50° F. (9° or 10° C.). The gases are further purified from naphthalene, sulphuretted hydrogen, and cyanogen. A standard washer was fixed between the coolers and the exhauster for the absorption of the naphthalene. This washer was supplied with Röpert oil, an anthracene oil containing 4% of benzol, and by means of it the whole of the naphthalene was absorbed. The exhauster now pressed the gas through a Pelouze apparatus which retain the wash oil (which had been carried along with the gases), and afterwards through three dry purifiers. These were three cast-iron boxes, 23 by 23 feet (7 by 7 metres) square, and about 6½ feet (2 metres) deep, fitted with three layers of wooden gratings, on which the bog-iron ore was placed. The gas went through the individual layers of the ore, and in so doing lost through oxidation the sulphuretted hydrogen in sulphur and water, and the greater part of the cyanogen through chemical combination. An iron sulphate solution washer for the quantitative removal of cyanogen was not provided. The gas, which was now sufficiently purified, went into a gas holder 53,000 cubic feet (1,500 cubic meters) capacity, which equalized the smallest variations of heating value, and at the same time acted as an intermediate holder for feeding the distance gas-exhausters. The holder then pressed the gas through a gas meter to the distance ex-

hausters (Roots blowers), which pressed the gas through pipes 17,000 feet (5.2 kilometres) long, and about 10 inches (250 millimetres) in diameter, with a pressure of about 40 inches (1,000 millimetres) of water-gauge, to the gasometers at the gas supply station.

Several other types of by-product recovery coke ovens will be found described in Appendix II., as at the time of going to press with this section suitable drawings for illustration purposes were not available. The chapter will therefore be concluded with some valuable notes on

Cause and Effect in By-Product Coking, by "C.M.G.," in the Coking Section of *The Gas World* for April 1st, 1916. The writer refers to a passage in the discussion of Mr. Taylor's paper on benzol, read before a meeting of the Coke Oven Managers' Association in December, 1915 (which will be dealt with in a later chapter), and says:—

"The passage referred to was included in the remarks made by Mr. E. M. Myers, as follows: 'What he had expected was rather something about the manipulation of coke ovens in relation to the production of benzol. . . . He wondered how far these things were related to the manipulation of the coke-oven plant, to the occurrence of suction or back pressure, for instance, or to moisture in the slack or to the temperature of the ovens.'

"In a general way this covers the question of the relationship between cause and effect in by-product coking, and there are other points, in addition to those mentioned by Mr. Myers, in which the real relationship does not appear to be understood. The whole question of by-product coking is now being regarded from an entirely different standpoint from what it was a few years ago. From rule-of-thumb methods we progressed to the introduction of the chemist. Now we are going a stage further, and are introducing also the physicist. The complexity of the problems of by-product coking is such that there is ample ground for inquiry on the physical as well as the chemical side. In this article the writer proposes to discuss some of these problems on the lines of inquiry suggested by Mr. Myers.

"At the outset we must assume that coke is the principal product, and must have prime consideration. Our first aim, therefore, must be to attain the best conditions for the production of the best coke, and then, as far as may be compatible with this, introduce such modifications—by manipulation, as Mr. Myers puts it—as will enable us to obtain the maximum yield of by-products. It is, of course, impossible to lay down general rules which are applicable without modification or quali-

fication to any and every plant, but as the principle is the same, adaptation to local circumstances should not be difficult. The temperature of carbonization is perhaps the most important factor. It has frequently been stated that some classes of coal require to be carbonized at a much higher temperature than others. But, in the writer's opinion, there is very little difference in the final result, provided the temperature is high enough to drive off all the volatile matter in a reasonable time. In general, the time of carbonization should be proportional to the amount of volatile matter in the coal, and the author believes that an average oven temperature of about 800°C . gives the best all-round results. If the temperature in the ovens gets above $900\text{--}950^{\circ}\text{C}$., there is the risk of fusion of the oven walls, and also, at the higher temperature, there is more breaking up both of the tar and the ammonia in the upper part of the oven. With an excessively high temperature, and particularly in the case of a high volatile coal, there is a very rapid evolution of gas, and this tends to make the coke spongy, and also to break it up rather more. In gasworks practice a few years ago horizontal retorts were only partially filled. Gradually charges were increased and the free space in the retort diminished, and better results were obtained almost invariably by following out this practice. The same applies in coke-oven practice. In the long run it pays to work fewer ovens and heavier charges; but, of course, care is necessary in order to ensure that the charges are not made too big. Special means are adopted in most systems of ovens to prevent the overheating of the crown of the ovens; if the charge is made too big the upper portion will take an unduly long time to burn off, and so reduce the capacity of the whole plant. We know the effect of temperature on the yield of tar and ammonia fairly well. Above 600°C . the yield of tar decreases rapidly, whilst about 850°C . is the best temperature for the production of ammonia. Probably the usual coking temperature on most plants ranges between 750°C . and 900°C . in the oven. Now, there will not be a great deal of difference between the tar yield at 750° and 850°C ., nor will there be in the quality of the coke. Hence, if 850°C . is the best temperature for the production of ammonia—this is not difficult to determine by experiment—the best plan is to make this the average working temperature, and at the same time to work with heavy charges. As already pointed out, light charges means a large free space, with consequent overheating of the gas, thereby decomposing both tar and ammonia, and bringing about a decreased yield of both.

“We are very much in the dark with regard to the effect of moisture on the various products, and the problem is rendered much more difficult by the fact that we are not able to dissociate the effects of

moisture from those of temperature and other factors. In order to determine the most suitable degree of moisture we have to look at the case from two points of view—(1) the effect on the general working of the plant, and (2) the effect on the coke and by-products. In these days of direct recovery the moisture content is of more importance than it used to be. With the semi-direct or cold processes the whole of the added moisture, together with the water of distillation, is thrown down in the coolers, and forms the ammoniacal liquor, which is subsequently distilled. The more moisture in the coal the more liquor there is to distil, and consequently the more effluent liquor there is to dispose of. As, however, facilities are provided for dealing with the liquor, it is not a very troublesome matter to the coke-oven manager, from the ammonia point of view: his chief concern is to dispose of the effluent. With the real direct or hot recovery processes, however, the matter is much more important. Here there are no coolers to eliminate the excess moisture, so it has to be kept in a state of vapour in the gas. Any which condenses necessarily carries with it a proportion of ammonia, and if no stills are employed the ammonia is lost. A close study of vapour tension at different temperatures is therefore very desirable, as only by this means can the amount of moisture in the gas be controlled. It will generally be found that if the moisture in the coal, together with the water of distillation, exceeds about 10% the temperature of the gas must at no point fall below 70° C., as otherwise a considerable proportion of liquor will be deposited. Now, if compressed coal is being used the total water is usually not less than 12 to 15%, so that some liquor is almost invariably produced. In many cases this is partly evaporated, any free ammonia being led back into the gas, whilst the fixed ammonia is obtained principally as a solution of ammonium chloride. It has been suggested that the addition of a proportion of lime to the coal will have the effect of fixing the chlorine in the coke, and so of decreasing the proportion of fixed ammonia which has to be dealt with in this way.

“With regard to the effect of moisture on the coke and by-products, a very complex series of possibilities is presented. Excessive moisture means unnecessary cooling of the ovens, and consequently uneven heating and longer carbonizing periods. With uncompressed charges, probably a maximum of 5% is near the best limit, whilst with compressed charges the limit should be the minimum for the production of suitable cakes which can be charged without breaking at all. With excessive moisture the coke is small and the production of breeze abnormal. Moisture has a protective influence on both tar and ammonia, in that it cools the free space above the charge, and so prevents to some

extent the decomposition of both these products. This protective influence is, of course, limited to the first half of the carbonizing period. Roughly speaking, it takes about an hour for each 1% of water to be driven off. The drying action is naturally from the sides to the centre of the charge, so that whilst the outer layers of the charge are giving off good gas the inner layers are only giving off steam. As long as any steam is being evolved, just so long will the protective action ensue. We know that the best gas is given off during the first half of the carbonizing period, *i.e.*, while the protective action is being exerted. It has also been shown that the illuminating power—which, of course, is an index of the richness of the gas—is higher for gas made from wet coal than from the same class of coal in the dry state. It might, therefore, be argued that the more moisture the better would be the results from the by-product point of view; but this is not so. Apart from the fact that the coke suffers—and this, after all, must be the deciding factor—there is no advantage in a large amount of moisture. The protecting influence is exerted for a longer period, but the time of carbonization is also prolonged, so that in the end nothing is gained, there being little improvement in the ratio of the protective period to the carbonizing period.

“ The effect of suction or back pressure has also to be considered from two points of view:—(1) The effect of suction or pressure on the purely chemical reactions which take place in the decomposition of the coal, and (2) the loss of gas due either to combustion through over-suction or escape through over-pressure. With regard to the first of these we are again comparatively ignorant. The reactions involved in the destructive distillation of coal under normal conditions are only very imperfectly understood, and if we modify the conditions we are hopelessly at sea. Many unexplainable reactions are now put down to catalysis in some form or other. With some catalytic reactions, increase of pressure accelerates the reaction, with others it causes retardation. In general, however, the variation in pressure required to produce any substantial effect is rather considerable. In coke-oven practice we are precluded from working with much pressure in the ovens, as otherwise the joints round the doors would blow, and also there would be escape of the gas into the flues. On the other hand, with over-suction air is drawn into the ovens, with consequent combustion inside, and its attendant troubles of hot ascension pipes and pitch in the main. Except in so far as it is a matter of direct loss through leakage or combustion, in the writer's opinion the effect of suction or pressure is, owing to the limits between which we are necessarily confined in ordinary coking practice, comparatively negligible. There is a system of low temperature car-

bonization which works under a very high suction, but no very striking results are attained thereby.

"In the matter of benzol production opinions differ as to what are the best conditions. It is generally accepted that the aromatic hydrocarbons are polymerization products, and also, generally, that such polymerization increases with the temperature. This implies, therefore, that the yield of benzol increases with the temperature. In the case of ammonia we are able fairly easily to determine a temperature-production curve owing to the ease with which ammonia can be estimated. Unfortunately, we have no simple test for the determination of benzol. Gasworks experience shows that naphthalene is a high-temperature product, and we know that benzene can be synthesized from acetylene at a high temperature. We can construct hypothetical equations for the production of benzene from various paraffinoid bodies, so that it seems quite likely that benzene is a high-temperature product. But is there a maximum limit, as in the case of ammonia? It is too complex to reason out on theoretical grounds alone. The writer's experience is that, within ordinary working limits, the higher the temperature the higher the yield of benzol. On the other hand, we have advocates of low temperature carbonization claiming for their system large yields of benzol and toluol.

"The whole question, therefore, of cause and effect in by-product coking is extremely complex, and a multitude of factors, all operating at the same time and in varying directions, are to be taken into account."

The Utilisation of Waste Heat from Coke-Ovens for Power Production.—Some notes on power production from the waste heat of coke ovens are contained in the "Transactions of the Institution of Mining Engineers," Vol. xxxix., Part 3, from which the following is quoted:—

"Whilst the various types of plant already mentioned are suitable only for non-caking coals, another important aspect of colliery power production for those districts which yield a good caking coal is that of coke-ovens; that is to say, the installation of ovens the primary object of which is the conversion of duff-coal into coke. The by-products of coke-ovens consist of gas, sulphate of ammonia, and coal-tar. The gas produced has a very high heat value, namely, from 400 to 460 British thermal units per cubic foot. In regenerative ovens about a half of the total gas given off in the process of coking the coal is consumed in continuing the distillation, leaving a surplus of about 5,000

cubic feet per ton of coal treated, which, after proper cooling and scrubbing is available for use in gas-engines or furnaces.

"There are now many installations of this kind at work in British collieries, no fewer than thirty sets of various types of retort coke-oven having been put down during the past two years in the South of Scotland and North of England alone. Mr. E. M. Hann has recently read an interesting paper* giving particulars of the coke-oven plant at the Powell-Duffryn Steam Coal Company's Bargoed Colliery, where gas-engines aggregating 6,000 horse-power are now at work.

"For purposes of comparison with the scheme for non-caking fuels just put forward, it might be mentioned that a coke-oven installation for the same gas-power duty would require ovens capable of dealing with 151,200 tons per annum. The yield of sulphate is low in coke-oven practice as compared with Mond plants, due to the high working temperature, which has the effect of destroying the ammonia present in the gas. Thus, 25 to 30 pounds per ton of dry coal treated would constitute a fair average, whilst each ton of fuel also yields about 80 to 90 pounds of tar. At £11 10s. and 18s. per ton respectively, these by-products (taking the mean figures) would bring in a revenue of £26,510 per annum. As modern coke-ovens have a coking efficiency of 75% (86% is not unknown on the amount of dry coal burnt, 113,400 tons of coke are thus available, which at 15s. per ton would yield £85,050. The total revenue (exclusive of gas) is found to be £111,560 per annum.

"The capital cost of such a scheme planned on a liberal scale would be about £130,000, including ovens, recovery plant, gas-engines, electrical generators, buildings, foundations, and accessories. Interest, depreciation, and insurance at 15% would amount to £19,500, whilst the complete labour charges of the installation amount to 1s. 3d. per ton of coal treated, or £9,450. Assuming the market value of gum-coal (caking) to be 6s. per ton, and adding £4,300 for acid, stores, and renewals, the total cost of production amounts to £78,610 per annum.

"From this it will be seen that not only is the whole of the power obtained free of cost, but there is the substantial profit of £32,950 per annum from the undertaking.

"If this scheme in turn be compared with a colliery steam-plant generating power at 0.35d. per Board of Trade unit, with an output of 21,600,000 units per annum, the net gain is seen to amount to £63,950 per annum on an outlay of £130,000, thus showing a return of about 50% on the capital invested.

"The heat possibilities of gas-engine exhaust do not yet appear

* "A Recent Plant for the Utilisation of Small Coal," by E. M. Hann, *Proceedings of the South Wales Institute of Engineers*, 1909, vol xxvi., page 199.

to have been fully appreciated. Special exhaust-heat boilers are available, and many of them are in use in connection with large gas-engine installations in this country, with such an efficiency that 2 to $2\frac{1}{2}$ pounds of steam per brake-horse-power-hour may be obtained at 130 pounds pressure, without placing any appreciable back-pressure upon the engine. Thus, an installation working at an average load of 3,000 kilowatts, as assumed in the foregoing estimate, would evaporate about 9,000 to 11,000 pounds of water per hour, which would easily drive a steam-engine continuously at a load of 625 horse-power, allowing 16 pounds of steam per indicated horse-power hour. This is waste heat converted into useful work, as in the case of the exhaust steam turbine.

"In the light of facts such as the foregoing, it may be wondered why British colliery owners and power users in general have been so slow to take up the large gas-engine, but no satisfactory answer is yet forthcoming. Great strides have been made in this matter in Germany, Belgium, and the United States, and large numbers of instances may be quoted in which units of 1,000 horse-power and upwards have been installed with most satisfactory results. In one case, that of the Indiana Steel Company of Gary (Indiana), there is an installation of 63,750 horsepower, consisting of seventeen Allis-Chalmers engines of 3,750 brake-horse-power each, whilst installations of 30,000 and 40,000 horse-power might also be mentioned.

"It does now appear, however, that the day of the large gas-engine in this country has at length arrived. Several large sets are at work in the Clyde, Cleveland, and South Wales Districts, together with an installation of 20,000 horse-power at Messrs. Brunner, Mond, and Company's works, Northwich, and one of 10,000 horse-power at the works of the Castner-Kellner Alkali Company, Limited, Runcorn. It is also worthy of note that one prominent firm of gas-engine makers is building and equipping a special works solely for the manufacture of large vertical gas-engines in units up to 3,000 horse-power.

"Actual experience has undoubtedly proved this system of gas-power production to be convenient, economical, and reliable, and there are substantial reasons for believing that, as the possibilities of the system become more fully understood, its application will be increasingly extended. A gas-driven central electrical power-station for a group of collieries is the ideal arrangement, where circumstances admit of the outlay which such a scheme involves, and there appears to be no doubt that many such will spring into existence during the next few years, with a view to using to the best advantage the heat possibilities of the coal consumed."

CHAPTER III.

GAS PRODUCERS.

The Crossley Suction Gas Producer.—The latest types of suction gas plant as manufactured by Messrs. Crossley Brothers, Limited, of Openshaw, Manchester, are shown in Figs. 64 to 70. Three distinct designs of suction gas producers have been brought out by Messrs. Crossley Brothers, the first two on the lines of the closed hearth plants, the latest design, introduced three or four years ago, being of the open hearth type. Fig. 64 shows a section of the new type Crossley plant, and Fig. 65 is from a photograph of a 450 B.H.P. plant of this type. Whilst the closed hearth type plants were and are satisfactory for generating gas for power and heating in those cases where the best grade of anthracite is obtainable, and where average running conditions exist, there were other cases where the use of inferior anthracite and gas coke was attempted, and trouble was experienced by the difficulty of removing the ash from the plant without risk of stoppage of the engine to which it was connected. The same difficulty presented itself where it was necessary to run a plant night and day for some considerable period without a stop. The closed hearth plant often causes difficulty in fulfilling this latter condition, and it has been the custom of some firms to use a duplicate generator, so that the engine could be changed over from one generator to the other without stoppage to allow of the removal of ash from the generator. Referring to Fig. 64, the generator (A) is a simple cylindrical steel casing, lined with fire-brick, supported by cast-iron legs (B), which also carry the circular plates forming the stepped grate, and provided with a fuel container and feeding hopper at the top. The necessary steam is raised in vaporisers entirely separate from the generator. As there are no vaporisers

at the top of the generator, it is possible to place a series of poker holes (r) on the top plate, adjacent to and parallel with the brick lining. By this means it is possible to remove all the clinker from the firebrick lining without in any way injuring the lining. Some classes of fuel form an excessive amount of clinker, and unless this clinker is removed from the sides of the generator, the quality of the gas made becomes poor, and

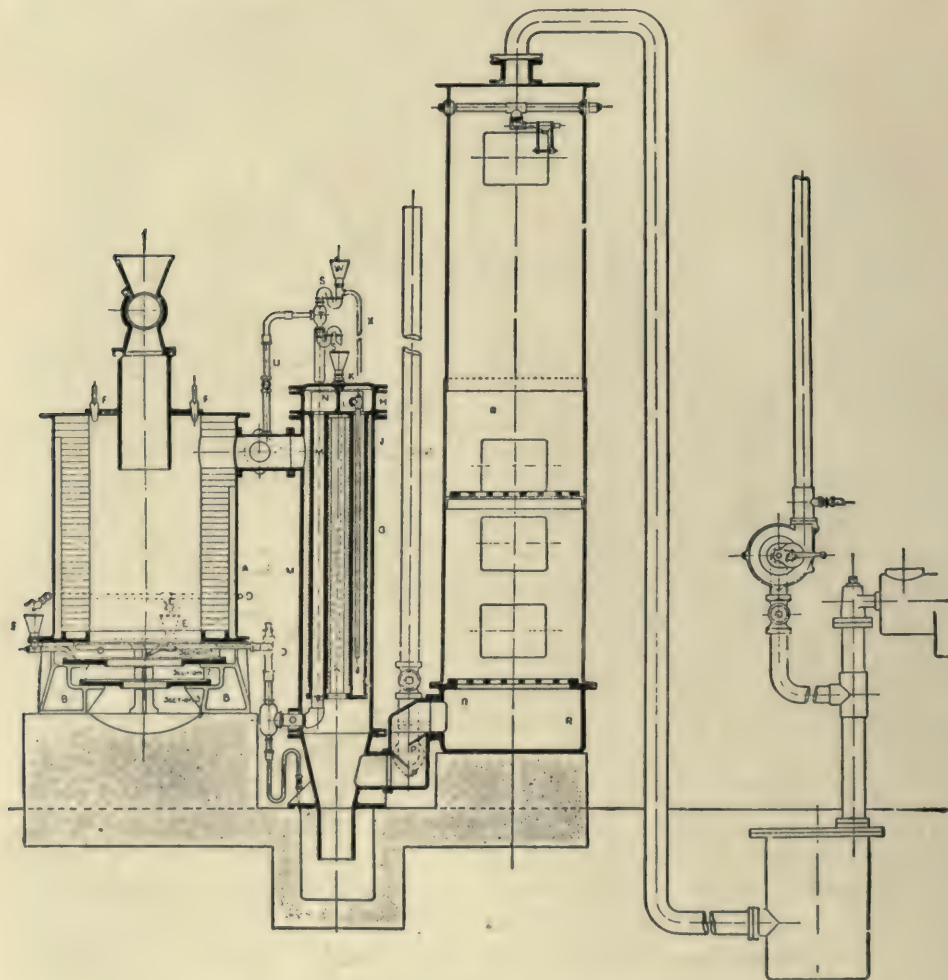


FIG. 64—SECTION OF CROSSLEY SUCTION GAS PLANT.

the engine is not able to carry its full load. In plants with the vapourisers in the generator the poker holes are usually placed at an angle in the fuel container, and when poking at an angle the attendant is frequently unable to tell whether, when poking, he is striking hard clinker which should be removed or the fire-brick. In the generator illustrated

the poker passes through the fuel parallel to the brick-work, and the attendant knows that any obstruction he meets is caused by clinker. This arrangement, therefore, makes it possible to keep the producer in

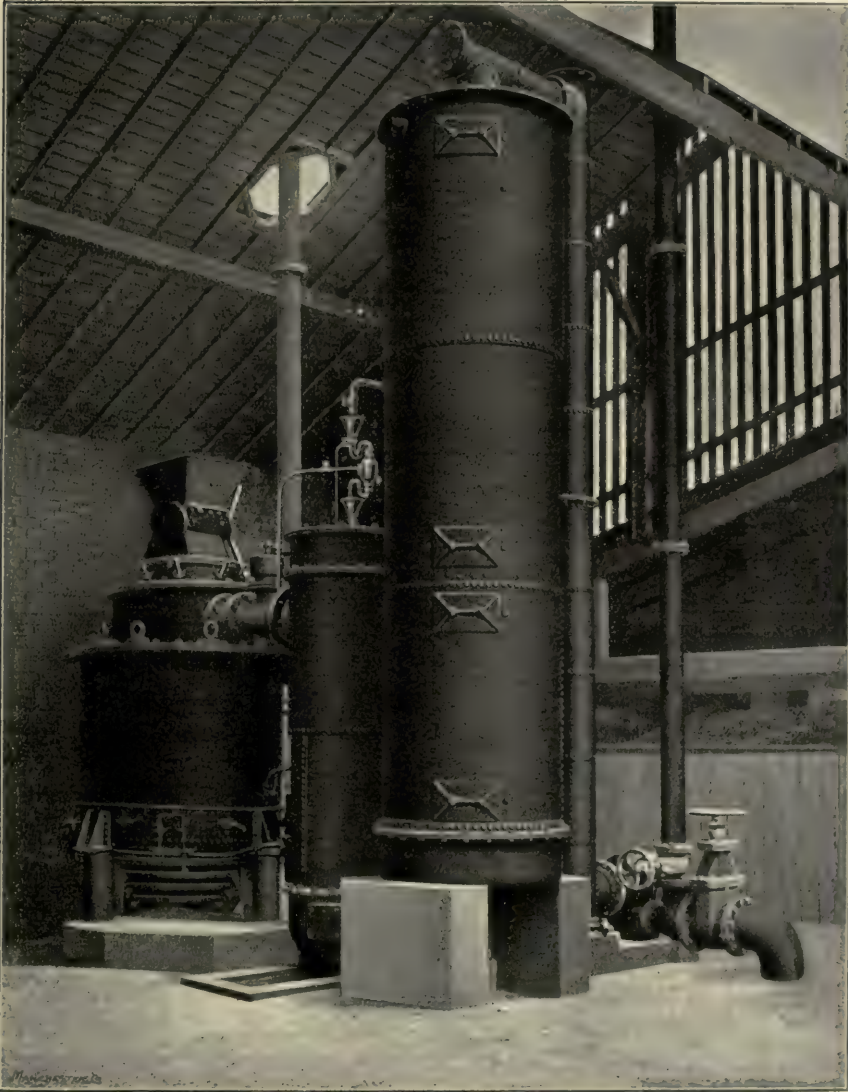


FIG. 65.—450 B.H.P. CROSSLEY SUCTION GAS PLANT.

first-class condition, and ensures that the gas made is of the right quality. The life of the fire-brick lining is also greatly prolonged.

It is, however, not sufficient to simply remove the clinker from

the fire-brick lining. Such clinker, together with the ash, must be removed from the generator itself, where long and continuous runs are necessary, otherwise the fire-grate would become choked. It is difficult for the ordinary gas plant attendant to do this removal on the ordinary flat grate type suction gas plant. Such generators are of the enclosed type. Fire doors must be opened to get at the ash and clinker, and the opening of the fire doors destroys the proper working conditions of the plant; the air, instead of being drawn through the vaporiser, where it mixes with the steam, is drawn directly through the fire door when this is opened, so that the steam supplied is altogether cut off from the fire. As a consequence of this, the quality of the gas falls, and the engine slows down, unless the fire door is quickly shut. Another disadvantage of this type is that when the door is open for cleaning purposes, it is practically impossible to remove the clinker, for as soon as any ash or clinker is drawn from one side of the fire, the fuel immediately above it drops down to take its place, and all subsequent efforts to remove ash and clinker result in a quantity of unburnt fuel being drawn out also. In practice, with the flat grate closed type of plant, the only portions of the grate that are really cleaned are those immediately adjacent to the fire doors. All these disadvantages are claimed to be entirely overcome by the new open hearth type plant.

The plates composing the stepped grate are so disposed as to lie outside the angle of repose of the fuel, which ensures that none of the fuel will of itself fall from the grate. Below the bottom plate the fuel rests on its own bed of ashes. The fire-grate is not enclosed, and the state of the fire can be examined without opening any fire doors, or otherwise interfering with the quality of the gas. Any clinker which has been poked down from the fire-brick lining, or which has formed on the grates, together with the ash, can be easily removed. Gas-making is not prejudicially affected, and the plant is able to work for long periods without the fire being drawn. All parts of the grate and of the generator can be kept quite free from clinker, and the section of the stepped grate plates is also made suitable for the efficient distribution of water, which is obtained from a small pipe running round the generator casing. This water passes in a minute stream into the funnels (E), and thence to the grate plates, overflowing from the upper to the lower. By this means the grate is prevented from being burnt out, and steam is quickly generated for gas making.

It is essential that no air be allowed to leak into the generator when feeding fresh fuel, and the charging valve shown in Fig. 66 has been designed to meet this condition. If any air enters the generator, other than through the fire bars, the gas is partially burnt in the gener-

ator instead of in the engine cylinder, thus causing increased consumption of fuel, overheating of the generator, and irregular working of the engine.

As will be seen from the illustration, a hollow cylindrical valve is employed working in a sleeve formed by the feeding hopper, and operated by handles. This design ensures an air-tight arrangement, and one which can be easily taken apart for cleaning purposes. Provision is also made to take up any wear which may take place on the valve seat. The design of the hopper is such as to render it impossible

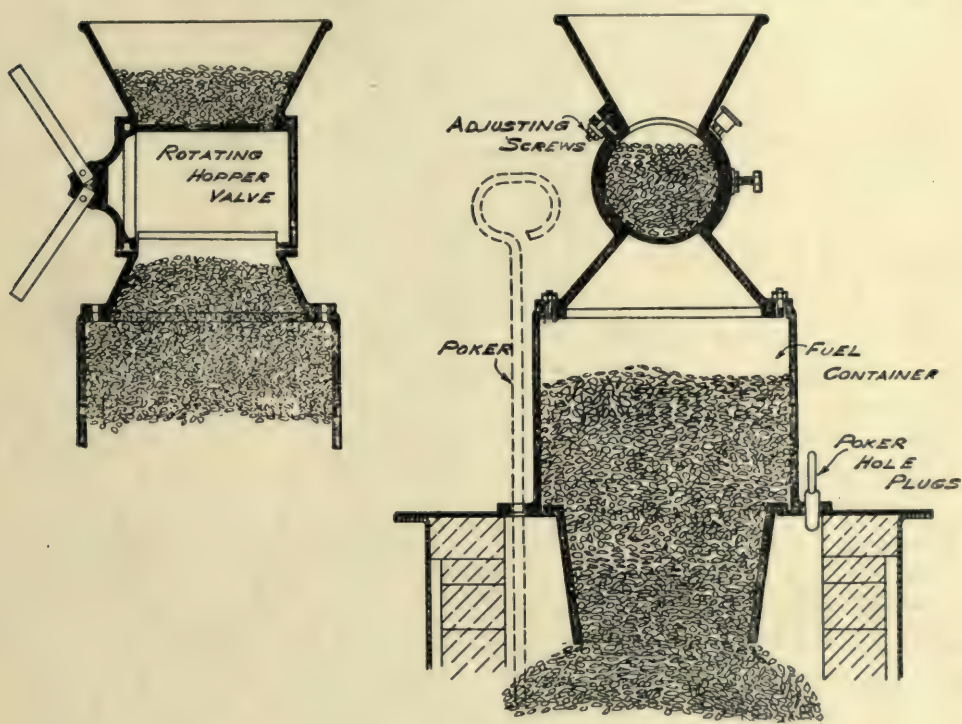


FIG. 66.—CHARGING VALVE OF CROSSLEY SUCTION GAS PRODUCER.

for the attendant, by forgetfulness or carelessness, to allow of air being drawn into the plant when charging.

From the generator the gas passes to the external vaporiser (g), (Fig. 67) which forms the communication between the generator and the scrubber. The vaporiser consists of an empty case, upon the top of which is bolted the box (H) carrying the gilled tubes (j). These gilled tubes have a maximum heating surface, and convey the heat extracted from the gas to the water which is to be vaporised. This water is first passed through the automatic water regulator (s), and flows down the

tube (κ). This tube is open at the bottom, and the water rises in the gilled tube to the overflow (ι), to which the feed tube of the next gilled tube in series is connected, and so on through the whole of the gilled tubes with the exception of the last one. This tube (μ) forms the communication between the steam space (ν) of the vaporiser and the steam distribution pipe (ο) to the producer. The water overflow also from the adjacent gilled tube passes down this pipe (μ), and is flashed into steam. There is thus a continual circulation of the water down the centre of each gilled tube and up the sides through each in series, and steam is taken away by the gilled tube (μ) and the distribution pipe (ο), which passes round the fire of the producer and distributes the steam through a number of spaced holes. It should be noted that with this design of vaporiser the gas which has to be cooled is external to the water which has to be heated, so that the water is continually surrounded by a jacket of hot gas.

A proper steam supply for mixing with the air drawn through the generator is of great importance, as without it the fire cannot be maintained at the correct temperature, nor can the engine develop its full power. With an insufficient supply of steam the temperature of the fire becomes too high, and clinker is formed; on the other hand, too much steam damps the fire down, and owing to the formation of an excess of carbon dioxide the heating value of the gas is reduced. Where the engine is working at a constant load the water feeding arrangements present no difficulty, as once the supply is adjusted to the load, no alteration need be made. As, however, most engines work under varying loads, to get the best results arrangements should be provided for automatically governing the amount of water admitted to the vaporiser, according to the load on the engine, and a special feature of the Crossley plants is the automatic water feed, by means of which the amount of water fed to the vaporiser is regulated automatically to suit the load on the engine. The quantity of steam supplied to the plant is hence a direct ratio of the power required from the engine, and as the load varies so the steam varies, with the result that the quality of the gas is kept very uniform even under widely varying load conditions. The construction of the vaporiser is clearly illustrated by the line drawing (Fig. 67). Every part is easily accessible, and the arrangement of the water tubes is such that they can be very easily cleaned in case hard or impure water is used. To examine the tubes it is only necessary to break the joint of the steam connecting point at the bottom, take off the top cover, and lift the whole of the tubes out with the water distribution box *en bloc*.

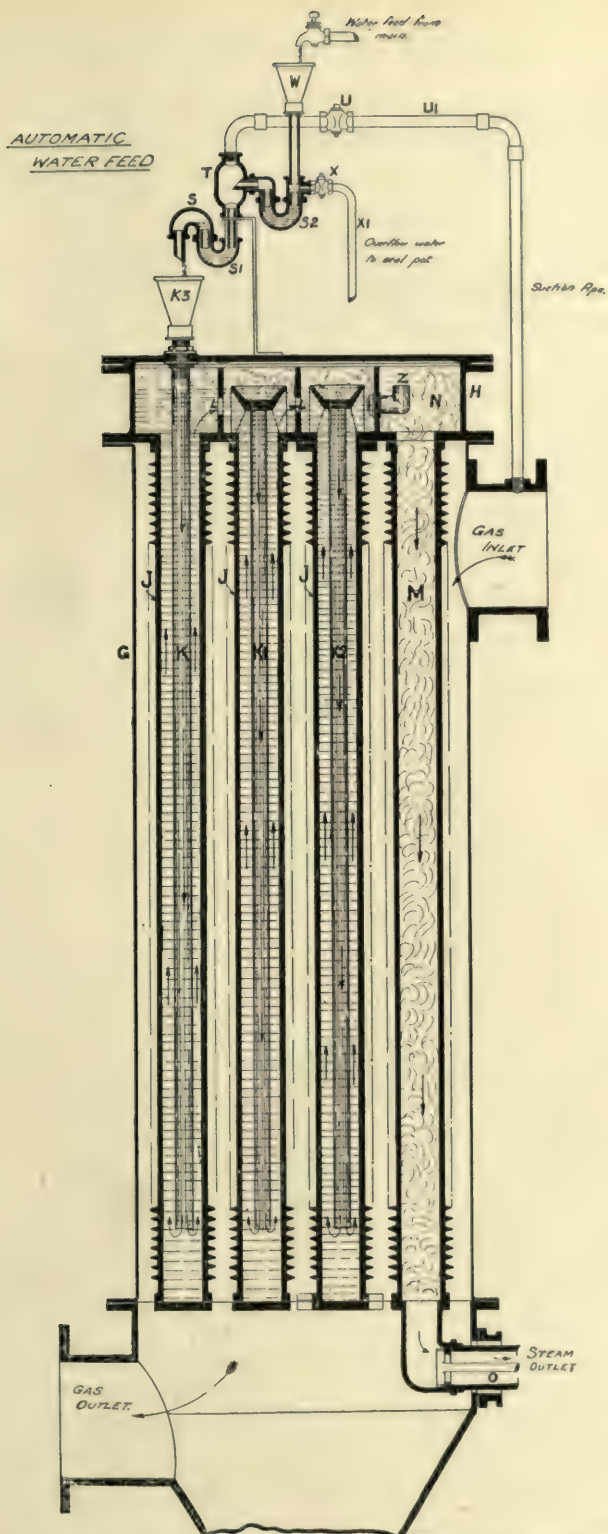


FIG. 67.—SECTION OF CROSSLEY VAPORISER AND AUTOMATIC WATER SUPPLY.

Each gilled tube can then be taken away separately and cleaned, and the makers state that the examination and cleaning of the whole plant can be carried out without even the withdrawal of the fire from the generator.

The construction of the vaporiser is further so simplified that one tube can be removed without interfering with the rest of the circuit water through the remaining tubes. Each of the gilled tubes is suspended from the water box by means of studs, the bottom end of the tube being free to expand or contract. The bottom end of the pipe is closed by means of a flange which is drilled to the same centres as the top flange of the tube, so that when a tube has been uncoupled for cleaning the bottom flange of the tube can be taken off, and the hole in the water distribution box at the top blanked up with this flange, and the vaporiser put back to its work whilst the tube referred to is being cleaned at leisure, the water passing through the rest of the circuit complete.

The water regulating arrangement consists of two tubes (s) (Fig. 67) coupled to a small vacuum chamber (r) which in turn is connected by the pipe (v) to the gas outlet pipe (v) from the generator. Water is fed from a tube into the funnel (w), and when the engine is not taking a charge of gas, this water overflows at the pipe (x) and runs to the seal box. When the engine takes a charge of gas, however, a vacuum is created in the vacuum chamber (r), and water is sucked over the upper tube (s) and runs into the lower funnel, and then to the vaporiser. There are no levers, diaphragms, or needle valves, and no moving parts.

Before going to the scrubber, the gas passes through two cascades of water (r) formed by the overflow water from the scrubber. The impact of the water on the gas frees it of the heavier impurities, which are continually washed away before it reaches the coke. The direct effect of this is that the work the coke scrubber is called upon to do is greatly reduced, and the coke does not require to be renewed so frequently as with other arrangements.

On leaving the generator, the gas, which in this type of plant has been considerably cooled by circulating round the external vaporiser, and by passing through the cascade scrubber, is still too hot to be used in the engine cylinder. It is essential that the gas be quite cool before it enters the engine, as otherwise the weight of the charge drawn into the cylinder is diminished, and the power developed by the engine thereby reduced. Unless the gas be thoroughly cleaned, the inlet valve on the engine is liable to stick, and the regular working of the instal-

lation is seriously disturbed. The scrubber is a vertical steel cylinder filled with coke, which rests on two trays. A water-spraying arrangement is provided at the top of the scrubber, which distributes the water evenly over the coke. The gas, which enters the scrubber at the bottom portion comes into intimate contact with the wet coke, and is cooled and cleaned. The base of the scrubber (R) is made of cast-iron, which has a longer life for this portion of the plant than mild steel. The coke, as mentioned previously, rests upon two trays. That contained on the bottom tray fouls much quicker than the coke on the upper tray. Filling and emptying doors are so arranged that the bottom portion can be removed without interfering with the coke in the upper portion of the generator. From the scrubber the gas passes to a combined drier and expansion box.

Instead of blowing up with a pressure fan, an exhaust fan is used, which draws the air and vapour through the fire, and in fact acts on the plant in an exactly similar manner to the engine, creating a vacuum in the pipe and the various portions of the plant, and producing rapid combustion in the generator. From the fan a blow-off pipe is led through the engine-house roof. The advantages of this arrangement are that the fan can be placed near to the gas engine cock, instead of in the gas plant house, so that when the operator has blown sufficiently long to obtain good gas, which is found by lighting the gas at the small test cock, he can at once start the engine, without having to give any instructions to an assistant at the plant, which may be some distance away. One man is sufficient to start the engine and plant. Also, with the pressure fan working on an ordinary suction plant, the conditions are quite different from those under which the plant is working when the engine is running, and there is a difficulty in getting the necessary amount of steam through the fire. The exhaust fan, on the contrary, draws in the steam, and the conditions are exactly the same as when the engine is working. As a consequence of this, the plant is very quickly started, saving both labour and fuel. A suitable seal box is arranged, either in the form of a cast-iron box, or as a well in the concrete for taking the water overflow from the scrubber, and for the convenient removal of any dust deposited in the connecting pipe between the generator and scrubber.

As regards fuel consumption, the plant described is designed to use anthracite or gas coke. Under normal working conditions the consumption of good Welsh anthracite is stated not to exceed from $\frac{7}{8}$ to 1 lb. (according to size of plant) per brake horse power hour at full load. The consumption of coke under the same conditions is from 1 lb.

to $1\frac{1}{4}$ lb. The fuel costs of working a 50 B.H.P. engine at full load are therefore as follows:—

| | | | | | | | |
|--------------|-----|--------|---------|-----|-----|-------|----------|
| With coke | ... | @ 15/- | per ton | ... | ... | 4d. | per hour |
| " " | ... | @ 17/- | " | ... | ... | 4.5d. | " |
| " " | ... | @ 20/- | " | ... | ... | 5.4d. | " |
| " anthracite | ... | @ 25/- | " | ... | ... | 5.8d. | " |
| " " | ... | @ 30/- | " | ... | ... | 7d. | " |

The water consumption in the vaporiser does not exceed 1 lb. per B.H.P. hour, and in the coke scrubber from $1\frac{1}{2}$ to 2 gallons per B.H.P. hour. Where the fuel used is of inferior quality, and a tar extractor is required, the water consumption is slightly increased.

A heat efficiency of about 85% is claimed by the makers for this type of plant, installations of which, they state, have been run for some months without stoppages of any kind. The facility for the removal of the ash allows of the use of inferior and smaller grades of fuel, which give it a wider scope and more economical power production. For fuels containing tarry matters, such as bituminous coal, lignite, wood and other refuse, it is necessary to use extra cleaning apparatus in order to get rid of the tar, and a patented centrifugal tar extractor has been effectively used for this purpose.

When the engine is stopped, the chimney cock on the gas outlet pipe of the generator is opened, and just sufficient air is allowed to pass through the fire to keep it alight. Compared with the heavy stand-by losses of the steam boiler, those of the suction plant are trifling. The coal consumed in a plant of 100 B.H.P. during an all-night twelve hours' stoppage amounts to less than 40 lbs., an amount which is less than a steam plant of the same power would consume in one hour.

Many attempts have been made to evolve a suction gas plant capable of dealing with non-caking bituminous fuels. This fuel is much more widely distributed than anthracite coal, and its cost is much below that of anthracite, particularly in those areas to which anthracite has to bear the expense of carriage. The wide variations of the analyses and physical properties possessed by bituminous coal have, however, been an effectual bar to its use in the ordinary suction gas plant. Bituminous coal contains a large but varying amount of tar, and generally a high percentage of ash, and unless the gas is effectually cleansed the tar is deposited in the gas engine, clogging up the valves and piston. The plant must, therefore, be designed so as to remove this tar thoroughly from the gas, and facilities must be provided for the easy removal of the ash and clinker whilst running. During recent years Messrs. Crossley Bros. have successfully installed a large number of these plants of various sizes, and their latest design of plant is shown

in Fig. 68. In this plant the cost of fuel for 50 B.H.P. per hour is stated not to exceed 4d. with non-caking bituminous coal at 10s. per ton

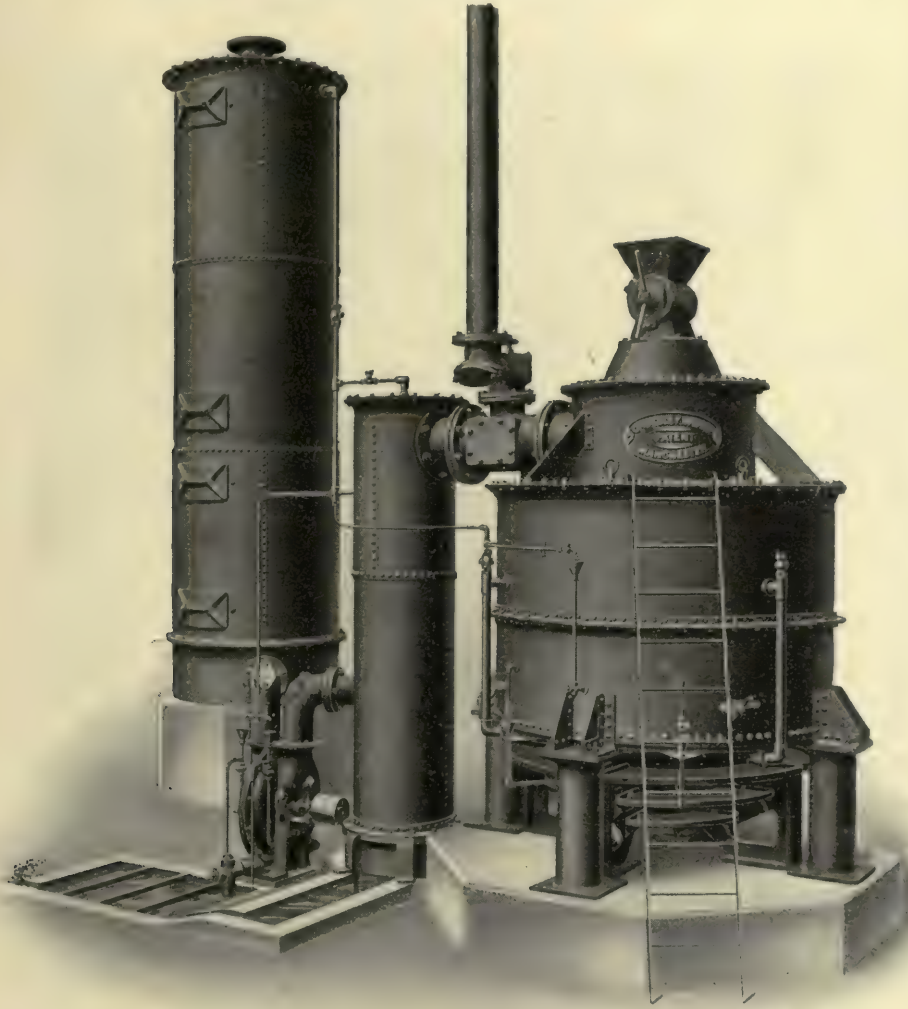


FIG. 68.—CROSSLEY SUCTION GAS PLANT FOR BITUMINOUS COAL.

A section of the Crossley pressure plant for non-caking bituminous fuel is shown in Plate IX. These plants are built in sizes from 250 B.H.P.; the plant shown has a capacity of 1,000 B.H.P. From the sectional illustration it will be seen that the producer is fed from the top by means of an air-lock hopper, an overhead bunker carrying a suffi-

cient supply of coal for an extended run, the fuel being raised from ground level by means of an elevator. The conical fire-grate is placed as low down in the producer as possible, so that its proximity to the

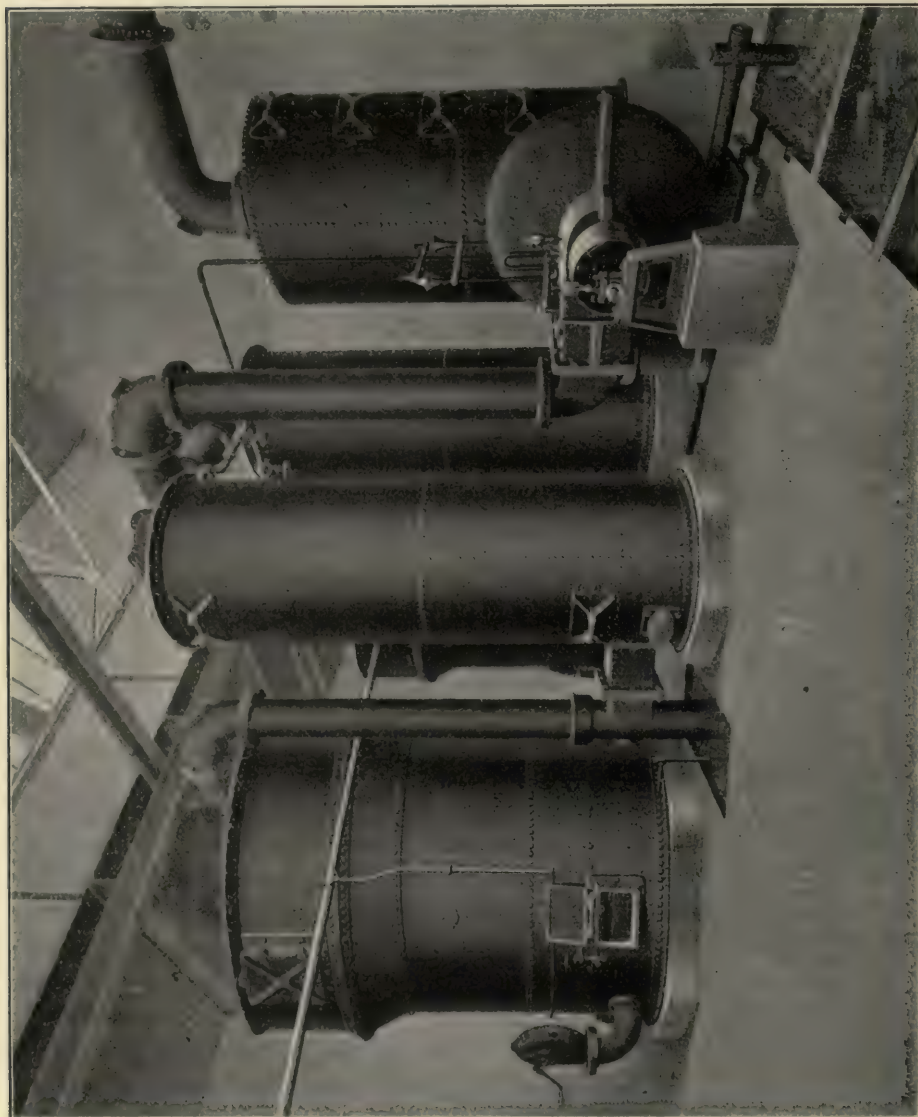


FIG. 69 — 350 B.H.P. CROSSLEY WASTE WOOD GAS PLANT.

water seal prevents warping and jamming. The blast pipe for the producer is taken below the water seal, so that there is no obstruction to the falling ash in the producer. The lower portion of the producer shell

Fig. 1. 1. 1. 1.

1. 1. 1.

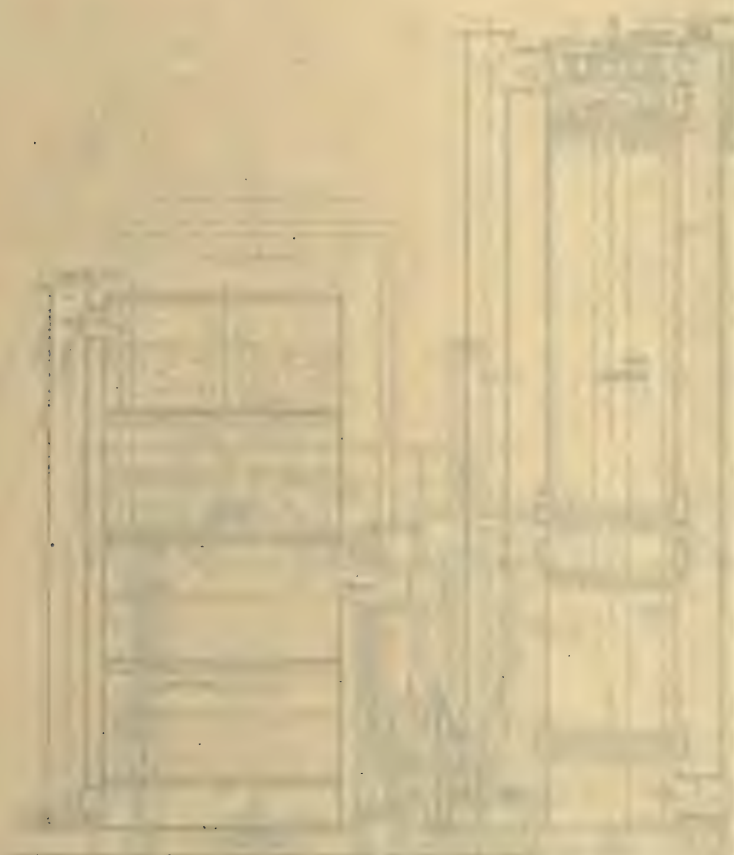


Fig. 1. 1. 1.

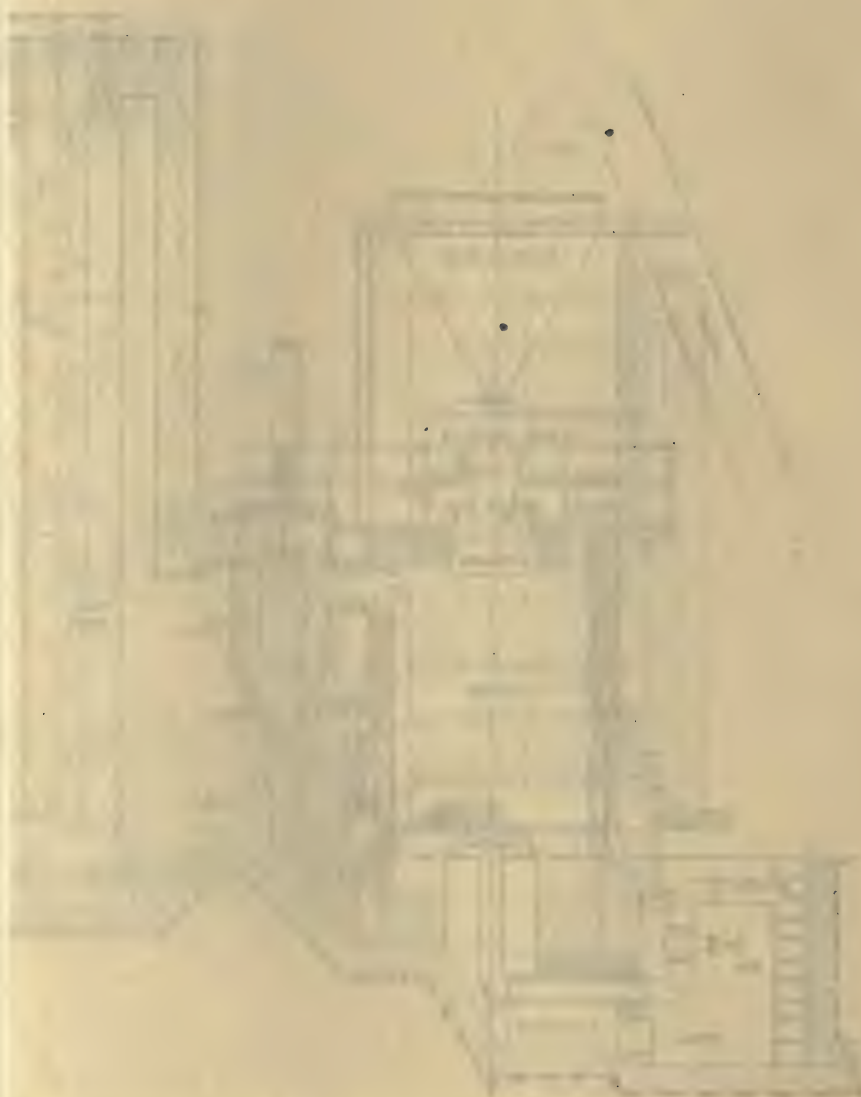


FIG. 1. A SIMPLE CHEMICAL PLANT LAYOUT.

is jacketed, and this being the hottest portion of the fire, steam is generated, and the deposit of clinker is obviated at this point. The gas as it leaves the producer passes through a dust-catcher on its way to the cascade washer. This cascade washer is a vessel divided into three vertical chambers; the passage of the gas is first down the outer compartment, then up through the centre compartment, which consists of the cascade proper, and afterwards down the other outer compartment on its way to the rotary tar extractor. In the scrubber the water is spread on the top inclined plate in a thin film and impinges on the lower plate, and so on from one to another, passing away at the bottom, with the impurities it has collected, into the sump. The gas passes through each cascade of water, and by this means most intimate contact between the water and the gas is obtained, the heavier impurities are washed out by impingement, and the plates are entirely self-cleaning. In the tar extractor the gas passes first to the centre of the machine, and is driven by centrifugal force to the periphery, where it passes at right angles to the stream of water thrown off from the propeller, leaving the machine at the centre on the opposite side to its entrance. Blades at each side of the impeller give a double-beating action. There is no pressure imparted to the gas by this machine, so that the power required to drive is only small compared with the amount of gas dealt with. From the tar extractor the gas passes to the coke scrubber, and thence to the wood wool filter, in both of which vessels it receives an extra filtering and purification. The plant illustrated, which is working in Birmingham, supplies gas for driving eight Crossley engines working night and day.

Fig. 69 shows a 350 b.h.p. waste wood gas plant, of which a large number are at work, gasifying waste wood refuse such as sawdust, chippings, bark, wood blocks, etc. A special air-lock hopper (Fig. 70) is fitted to this producer, suitable for all grades of fuel, and affording facilities for working with varying depths of fuel bed.

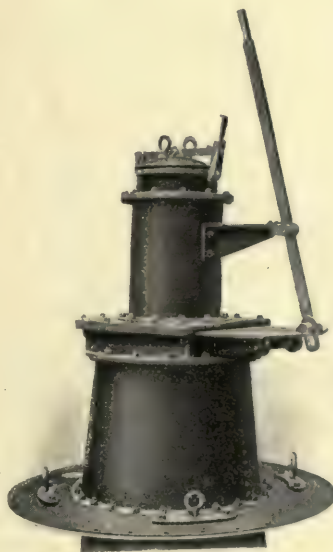


FIG. 70.—AIR LOCK HOPPER,
WASTE WOOD PLANT.

The Mond Gas Producer.—The development of progress in the design of by-product recovery gas producers is traced in a paper read by Mr. Arthur H. Lymm before the New York Local Section of the American Society of Mechanical Engineers. (For abstract of paper see *The Iron and Coal Trades Review*, May 28th, 1915). Mr. Lymm observes that "the early attempts in Europe to recover the by-products of the producer gas process are generally recognised to have been made in Great Britain. In that country the knowledge that the treatment of fuel by a mixture of steam and air (the former in excess) would convert a large percentage of the nitrogen contained in the coal into ammonia was first applied in practice on a large scale. The details of a plant to operate on this principle had been worked out by Messrs. Young and Beilby in England, and Grouven in Germany, among other investigators. The gas producer designed by Young and Beilby differed in operation from the ordinary by-product gas producer in that it was heated from the outside. The coal was distilled in the upper part of the producer or retort, and the tar vapours passed down through red-hot coke, and were (it was claimed) decomposed into permanent gas and ammonia. The coke in the lower half of the producer was burned in a mixture of steam and air, and the resulting gases, together with the gaseous products of the coal distillation, passed out of the producer by way of exits at the middle. It is particularly interesting to note that, as far back as 1883, Young and Beilby claimed to recover in the form of ammonia from 60 to 70% of the total nitrogen in the fuel. Although their retort was heated from the outside instead of the air and steam blast being superheated, it will be noted that the results claimed by them as to ammonia were not far short of what we realise to-day. The gas composition, too, was practically the same as that of the gas which has since become so widely known as Mond gas, and which has the following percentage composition:—Carbonic acid, 14 to 16%; carbonic oxide, 10 to 12%; methane, 2 to 3 %; hydrogen, 25 to 29%; nitrogen, by difference. It is now rather more than twenty-five years since the late Dr. Ludwig Mond first put into commercial practice the process, described in his British Patents No. 3821 of 1883 and 8973 of 1885, of gasifying fuel by means of steam and air, and simultaneously recovering the ammonia."

The text of Mond's Specification No. 3923 of 1883, for "improvements in obtaining ammonia, tarry matters, and combustible gases from coal or other combustible nitrogenous substances," is as follows:—"Numerous inventors have attempted to obtain ammonia by burning coal or other nitrogenous substances with a limited supply of air in the presence of steam. They have generally supposed that the steam and

fuel ought to act upon each other at high temperature, so that the incandescent carbon should decompose the steam and produce nascent hydrogen, which it was believed would combine with the nitrogen present in the gases and produce ammonia, and have therefore invariably worked their furnaces at a bright red heat. I have demonstrated by a long series of experiments that nascent hydrogen and nitrogen do not form any ammonia under those circumstances; that all the ammonia obtained by the treatment proposed is derived from the nitrogen existing in the fuel, and that only a small proportion of this nitrogen, say from one-seventh to one-fifth, can thus be obtained in the form of ammonia. I have, however, found that if the fuel be burnt nearly the whole of the nitrogen contained in it is easily obtained as ammonia if the fuel is burnt in the presence of steam at so low a temperature that the ammonia once formed is not dissociated again, namely at a dull red heat. In this case the tarry products are also of higher value, while the combustible gases obtained do not suffer in heating power. In order to maintain this low temperature throughout the furnaces or gas producers in which the fuel is treated, I introduce with the limited supply of air a large amount of steam, or preferably I load this air with water atomized by spray apparatus, or I may use both steam and atomized water. The gas formed by this mode of working differs considerably from ordinary producer gases; it contains a comparatively small amount of carbonic oxide and large quantities of carbonic acid and hydrogen which have been produced by the action of the carbonic oxide upon the vapour of water. As the carbonic oxide is atom for atom replaced by hydrogen, the heating power of this gas does not suffer, but is slightly increased. In order to collect the ammonia and tarry substances, the gases are passed through cooling and condensing apparatus in the usual way. I prefer to use the solutions of chloride of calcium or of magnesium, or sulphuric acid for absorbing the ammonia, as these allow the ammonia to be absorbed from the gases without complete cooling. It is also possible to keep the temperature of the burning nitrogenous substances sufficiently low by the use of air partly deprived of its oxygen, such as chimney gases or a mixture of such, or other inert gases with air, and in this case only a small amount of water or steam is required to obtain nearly all the nitrogen of the fuel in the form of ammonia. The gases resulting from this treatment are, however, very poor in combustible constituents, and consequently of little value. In place of steam or atomized water, or of air deprived of its oxygen, I may mix with the air a portion of the cooled combustible gases produced by the process, but while this plan will be found very effective when properly carried out, it is so liable to accident that I prefer at the present time the use

of atomized water with the limited supply of air. In conclusion, I am well aware that water or steam has been admitted into gas producers for many years past so as to make use of the surplus heat given off by the combustion of carbon into carbonic oxide, and that it has been also known that this steam aids the production of ammonia, increasing the quantity produced. But I believe no one prior to my application for this patent ever proposed to carry on the entire operation of converting carbonaceous fuel into combustible gases by means of air so largely charged with water, steam or indifferent gases that the temperature of combustion should not exceed the temperature of dissociation of the ammonia formed, namely a dull red heat, and therefore I claim (1) The improvement in obtaining ammonia, tarry matters, and combustible gases from coal or other nitrogenous combustible substance, which consists in converting the entire combustible matter into gases at one operation, by passing through such fuel air mixed with such a large excess of steam or water, or steam and indifferent gases, as to maintain the temperature of combustion at a dull red heat throughout the mass of burning fuel, whereby a greatly increased yield of ammonia is obtained. (2) The improvement in the manufacture of ammonia, tarry matters and combustible gas from nitrogenous combustible material, which consists in passing into the fuel air strongly charged with water spray or atomized water."

The following is the text of Mond's Specification No. 8973 of 1885:—

"In order to condense ammoniacal products and tar from producer or furnace gases, these gases have hitherto been cooled by passing them through iron conduits exposed to the air or surrounded by water. In either case this operation has been found very tedious, and has required enormous surfaces on account of the slowness with which the heat is taken up from the hot gases by the sides of a conduit through which these gases pass; and in the case of producer gases obtained under my Patents Nos. 3821 and 3923 of 1883, which contain a large percentage of steam, these difficulties have again been much aggravated by the large amount of heat stored up as latent heat in the steam, and which has all to be abstracted from the gas by cooling.

"To overcome this difficulty I have devised the following method: I pass the gases through a scrubber or series of scrubbers of any convenient construction, in which I bring them into direct and intimate contact with a quantity of water sufficient to reduce the temperature of the gases to below 40° C., and passing through the scrubbers in a direction opposite to the current of gas. The water issuing from these scrubbers has absorbed the ammonia and ammoniacal compounds, and has condensed the tarry matters, which remain suspended in it, but con-

tains both in very small quantities; it has also condensed the steam contained in the gases, and has become hot. This hot water I pass through a methodical cooling apparatus, through which it flows in one direction, while a current of cold water flows in the opposite direction. By preference I use wrought iron cylinders, in which a large number of wrought iron pipes have been fixed in the manner used in surface condensers. In these coolers I have a hot liquid on one side, and a cold liquid on the other, and am therefore able to pass a very much larger amount of heat through the iron tubes or plates separating the two liquids than I could if I had gas on one side and a liquid on the other, therefore very much smaller cooling surfaces will effect my object. The ammoniacal water issuing from the scrubbers after being passed through these coolers is again passed through the scrubbers, then again through the coolers, and so on; the same water being constantly used. On its way from the scrubber to the cooler, and from the cooler to the scrubber, the water is passed through a tank of sufficient size to allow the tarry matters to separate from the water. The tar accumulating in these tanks is removed from time to time.

"The steam in the producer gases being condensed by this treatment, the volume of water passing through the scrubbers gradually increases. A quantity of water equivalent to the condensed steam is from time to time abstracted. This contains a sufficient quantity of ammonia to make it readily available for the manufacture of ammonia or its salts by distillation or other well-known methods. The water used for cooling in the surface cooler takes up from the ammoniacal water the heat originally contained in the gases, including the latent heat of the steam contained therein, and thereby becomes hot. This hot water I utilise as hereinafter described.

"When I want to obtain the ammonia in the form of sulphate direct I pass the gases issuing from the gas producers into a scrubber or series of scrubbers, through which I pass a very dilute solution of sulphuric acid, not exceeding 2 of oil of vitriol to 100 of water. Other acids can of course also be used, such as hydrochloric or phosphoric acid, but I prefer sulphuric acid. These scrubbers are maintained by the heat of the gases at a temperature of about 100° C., so that no steam is condensed in them. The ammonia is absorbed in the scrubbers by the sulphuric acid, and the heavy portions of the tarry matters are condensed, and flow out of the scrubbers with the solution of sulphate of ammonia formed therein. I use the sulphuric acid so very weak, so that it shall not act on these tarry matters. If it is used stronger the tarry matters are dissolved by the sulphuric acid, and can no longer be separated from the solution of sulphate of ammonia. To

separate the tar, I run the solution through a sufficiently large vessel (separator) in which the tar accumulates. The solution of sulphate of ammonia issuing from this vessel is very weak, so that it would be very costly to utilise it by concentration or distillation. I add sulphuric acid to this weak solution, so as to bring it again to the original acidity, and then pass it again through the scrubbers, and I continue to do so until I obtain a nearly concentrated solution of sulphate of ammonia, from which the salt is then readily obtained in a marketable form, by crystallisation and evaporation.

"The gases after leaving these scrubbers are still at a temperature of 100° C., and contain a large percentage of steam, which has been increased beyond the original percentage by a certain amount of evaporation taking place in the acid scrubbers. These gases may, without inconvenience, be burned without previous condensation of the steam they contain. But in this case, the latent heat of this steam is entirely lost. In order to utilise this latent heat, I pass the gases after they leave the acid scrubbers through a scrubber or series of scrubbers in which they come into intimate contact with water passing in the opposite direction. In these scrubbers steam is condensed together with the lighter portions of the tar, and the water passing through them is raised to a temperature of from 90° to 100° C. This hot water (or the water which has been used for cooling in the surface coolers mentioned in the early part of this Specification, and has thereby become hot) I pass through another scrubber or series of scrubbers through which I draw in an opposite direction the air wanted for burning the fuel in my gas producers. This air thus gets heated and fully saturated with moisture at the temperature which it attains. If it reaches a temperature of 80° C. it will in this way take up sufficient moisture to make a further addition of steam unnecessary when using this air in the gas producer, and even at a temperature considerably below 80° the air will take up an amount of moisture which will very materially reduce the quantity of steam hitherto used at the gas producers. Thus all, or the greater part of the fuel now used for raising steam for the purpose of working gas producers under my Patents Nos. 3821 and 3923 of 1883 will be saved, and the latent heat of the steam once used is thus utilized to produce this steam over and over again.

"The tar which accumulates in the water used in these scrubbers is separated and removed from time to time. If any ammonia escapes absorption in the acid scrubber it will be absorbed by the water in the subsequent scrubbers. As the water heated in one of these scrubbers is cooled down in the subsequent scrubber, the same water may be used over continually. It will then gradually absorb more and more ammo-

nia, and may become sufficiently strong to make it worth while to recover this ammonia by distillation or otherwise. Throughout this Specification by the word 'scrubber' I wish it to be understood to mean any suitable apparatus used for bringing gases into intimate contact with liquids, such as absorbing towers, mechanical washers, spray apparatus, and the like."

Describing the original Mond producer, Mr. Lymm, in the paper already referred to, observes that it was rectangular in section, and was formed with a kind of double chamber. Its operation was similar to that of Young and Beilby, in that the coal was distilled in a downward direction in the upper part, and the coke residue was gasified in the lower part, all the gases mixing and leaving the producer together. The gas was passed into a long horizontal rectangular washer, and a fine spray of water was thrown into it by a series of revolving dashers. By this means a large proportion of the dust was removed, which was afterwards taken out of the water lute manually with long scoops, an irksome operation. From the washer the gas was conducted into a high lead-lined acid tower filled with earthenware ring tiles, where in passing upwards it came into contact with sulphate of ammonia solution trickling down. This solution contained a slight excess of sulphuric acid and deprived the gas, by absorption, of nearly all the ammonia contained in it. From this tower the gas was passed into a similar tower, called the scrubber, where it was brought into contact with cool water for scrubbing and cooling it, and it was then delivered for use.

The water, having taken up the heat of the gas, was collected in tanks, from which it was introduced into the heater, also of tower construction. It was here brought into contact with the cold air to be used in the producers, saturating this air with water vapour at from 70 or 80° C., and becoming again cooled. Then it was returned to the scrubber, where it again took up the heat of the gas, and so on in a continuous cycle.

After this plant had been in operation for some time, it was found that, owing to the large proportion of steam in the air blast, the heat value of the gas was below the desired standard, and also the yield of ammonia was less than that which Dr. Mond had set out to obtain. It was therefore decided to change the design of the gas-generating part of the plant, so that the air and steam blast would enter the producer with a considerable degree of superheat, thus enabling a still greater excess of steam to be used. This modified design was disclosed in Mond's British Patent No. 12,440 of 1893. The producer was made circular in section instead of rectangular, and its whole shell was surrounded by a jacket through which the air was passed on its way to

the grate, reducing the losses from radiation, and at the same time further superheating the steam and air blast. Directly contiguous to the producer was arranged a super heater, consisting of a series of parallel tubes with alternate ends connected, surrounded by a series of larger tubes forming an annular space. The gas from the producer passed through the inner tubes and superheated the steam and air blast, which was passed through the annular space in a counter-current direction on its way to the producer. With this provision, the gas was found to possess a much higher heat value, and a considerably increased yield of ammonia was obtained without extra fuel.

Fig. 71 is a reproduction from the drawing accompanying Mond's Specification No. 12,440 of 1893, in which the producer is thus described, the illustration showing a vertical section. *A* is the body or chamber of the gas producer, surrounded by *b b* brick lining, *c c* inner iron casing, *d d* outer iron casing, *e e* is a hollow iron platform to which both these casings are rivetted, and which allows unequal expansion of the two casings to take place without damaging the structure. *f f* is the top of the gas producer consisting of a single iron cover fastened and made tight to the inside shell, and lined with brick-work. *g g* is a bell-shaped casting which is always kept partly filled with fuel, and which is charged by means of a feed hopper into which the fuel is delivered from the reservoir *k*. *j* is a conical grate, *l l* is the water-lute, and *m m* the feet carrying the whole structure. *p p p* are the pipes or conduits for the producer gas, surrounded by *q q*, pipes of larger diameter, thus forming an annular space *s s* through which the mixture of air and steam is forced into the apparatus by a suitable fan or blower, entering through the pipe *r*, and passing round the pipes *p p p*, and subsequently through the annular space between the two iron shells *c* and *d* in the direction of the arrows until it arrives at the grate *j*, taking up heat all the way, and thus arriving at the desired temperature. The gas issuing from the producer carries with it some dust which is apt to settle in the pipes *p p p*, and requires removing from time to time. These pipes *p p p* are therefore arranged vertically in zig-zag form, with short horizontal branches between them, and are provided with blank flanges *y y* at their bottom to make them accessible for cleaning, and with a small opening *z z* (closed by a plug or cock) in these flanges through which the dust can from time to time be blown out without interfering with the work of the producer. To avoid loss of heat, the pipes *q q*, as well as the outer shell *d d* of the producer, are covered with a non-conducting composition. The inner shell of the producer ends in the casting *n*, which carries the wedge-shaped fire-bars (*i i i*) of the conical grate *j*, which are supported by a ring *o o*.

Mond's Specification No. 27,496 of 1903 relates to a gas producer having a circular grate in the form of an inverted truncated cone, between the bars forming the periphery of which cone the air, steam, or mixture of air and steam is supplied to the producer. The fuel hopper at the top of the latter has an extension projecting downwardly for a certain distance into the producer, and preferably of conical shape.

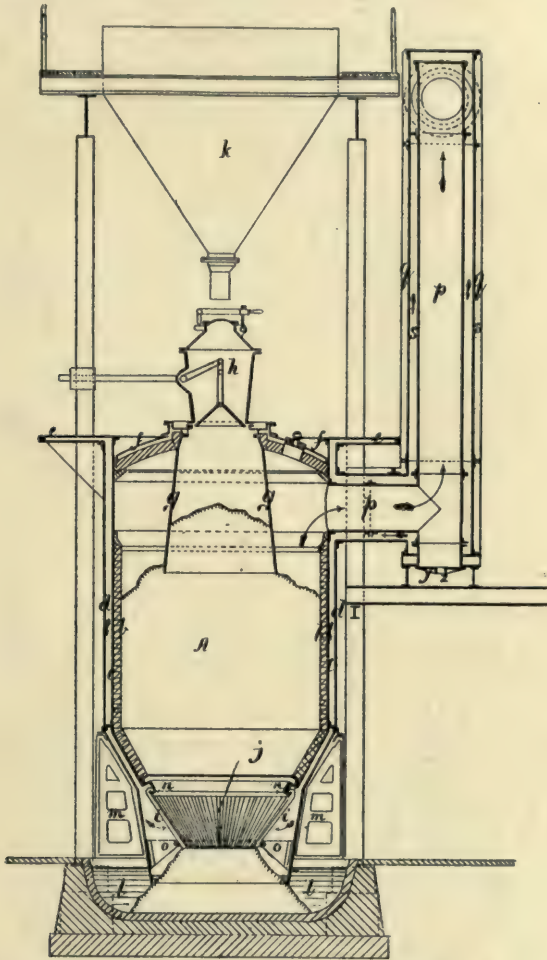


FIG. 71.—MOND GAS PRODUCER, BRITISH PATENT NO. 12,440 OF 1893

These features of a producer are described in Specification No. 12,440 of 1893. In the present form there is introduced into the lower part of the producer, preferably axially through the grate, a blast pipe for supplying air, steam, or a mixture of air and steam in addition to that supplied through the grate. The blast pipe should have a suitable hood to prevent fuel from entering it. The proportion of blast having access to the

producer through the pipe and grate respectively should be regulated, for which purpose the blast mains may be provided with valves or the like. The pressure of each blast may also be controlled by separate fans or the like. As in the producer described in the previous specification, the blast may be preheated by passing through a jacket surrounding the producer. Fig. 72 shows a producer constructed according to Patent No. 27,496 of 1903. The producer is practically the same as that described in Specification No. 12,440 of 1893, but is so arranged that the air, or air and steam blast which may or may not have previously passed through superheated pipes, enters the pipe *n* and is divided, one part passing through the annular space *a* between the two wrought iron casings *b* and *c* of the producer, as indicated by the arrows, to the bars *d*, while the other part passes through the pipe *e* to the tuyere *g*. The latter is covered with a hood *h*, which prevents any fuel from entering it. In order to obtain by this method producer gas of high quality it is most important that the proportion which the air or steam blast passing to the pipe *e* bears to that passing to the grate *d* should be accurately adjusted. For this purpose are provided the two valves *l* and *m*, or with the object of achieving this result more effectively, two different fans or their equivalent may be used to provide the necessary air or air and steam blasts, in which case the pipes *n* and *e*, through which the blasts are supplied to the tuyere and to the grate, would be independent of one another.

The claim of this Specification was: "(1) In a gas producer, the combination of a grate in the form of a truncated cone with a tuyere extending substantially axially therethrough, and devices whereby the proportion of air or of air and steam which passes into the producer through the tuyere to that which passes through the grate can be adjusted. (2) In a gas producer, the combination of a grate in the form of a truncated cone, with a tuyere extending substantially axially therethrough and two fans or the like, one supplying air to the tuyere and the other supplying air to the grate."

In Specification No. 28,031 of 1903, Dr. Mond says:—In my Specification No. 12,440 of 1893 I have described a gas producer having in its upper part an iron cylinder or truncated cone, which I shall term "a bell," reaching some distance down into the producer. This bell is always kept partly filled with fuel which after undergoing partial distillation in the bell, passes continuously into the combustion zone of the producer below. The products of this distillation in the bell have hitherto been compelled to escape at the bottom of the bell, and to pass through the incandescent fuel underneath it with the object of

converting the tarry matters they contain into permanent gases. With certain fuels I have, however, found that it is more advantageous to let these volatile products escape through openings in the bell, together with the gas generated in the producer, and thus to recover as large a quantity of tar as possible as a by-product of the gasification of the fuel. For this purpose, by my present invention I provide the bell with

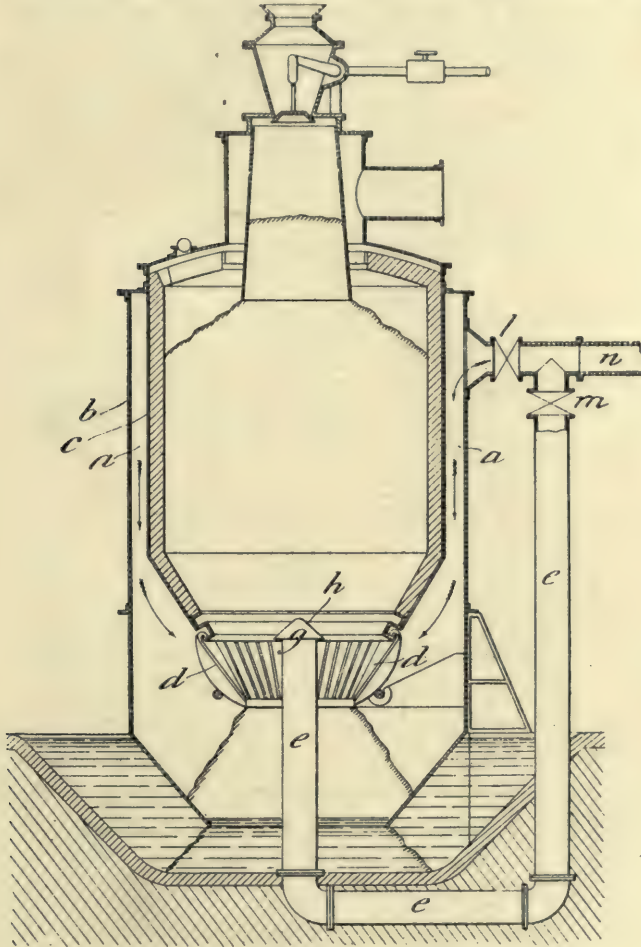


FIG. 72.—MOND GAS PRODUCER, BRITISH PATENT NO. 27,496 OF 1903.

one or several openings, slots or holes affording passages for gases and vapours, and preferably I arrange for the control of such passages from without the producer. When the passages are open a certain quantity of gas rises through the fuel contained in the bell, and escapes through them, carrying with it any tarry vapours evolved from the fuel contained in the bell. By adjusting the opening of these apertures I am able to

let more or less gas pass that way, thus regulating to a great extent not only the quantity of tar I wish to produce, but also its quality. When these apertures are open to their full extent a comparatively large quantity of gas goes through them, and the tar becomes much more fluid, whilst when they are closed the tar becomes stiffer, and approaches more nearly to the consistence of pitch.

Fig. 73 shows a vertical section through the bell and hopper of a producer having these improvements. The hopper *a* is of ordinary construction, and the bell *b* is similar to that described in the previous specification, except that it has holes *c* in it, provided, within the bell, with hoods *d*, and without the bell with covering slides *e*. The latter work in guides *f*, and are raised and lowered by hand wheels *g*, the bosses of which are nuts working on the screwed ends of rods *h* fixed to the slides. The gases escape through the holes *c*, when the slides *e* are more or less raised, into the casing *i*, whence they pass through the main *o* to the usual purifiers. The holes *c* may be more or less closed by flaps instead of by the slides *e*, such flaps being connected by links with rods that slide through stuffing boxes fixed to the casing.

Specification No. 6,719 of 1904 thus describes a further modification:—In Specification 12,440 of 1893 is described a gas producer specially adapted for converting ordinary slack or small coal into producer gases containing a minimum of tarry condensable matters, and obtaining as a by-product a maximum of ammonia. This gas producer is cylindrical in form, and provided with a grate of the shape of an inverted truncated cone through which the blast is admitted. In the top of the producer a bell is inserted reaching a considerable distance down into it, and this bell is always kept partly filled with the raw fuel, the volatile constituents of which are, to a certain extent, distilled off in this bell, and pass through and partly over the incandescent fuel, and are thereby converted into permanent gases. The fuel, thus deprived of a part of its volatile matter, passes continually into the producer, and part of it sinks down right underneath the bell to a considerable depth, forming an egg-shaped mass of dead fuel. If it is attempted to force the producer beyond its capacity, this mass of dead fuel may extend so far downwards that part of it may come out of the producer with the ashes. This is more particularly the case in large producers of considerable diameter, say about 10ft. This construction is specifically referred to in Specification 8,449 of 1901, which describes a modified process by which a portion of the hot gas produced in the body and bell of the producer is caused to pass back again with a certain portion of steam into the middle of the incandescent fuel at a level a little above the fire bars, it being also proposed that the fuel should be stirred by means of

arms in a manner analogous to that described in Specification 10,049 of 1900, which provides for the revolving stirring arms having not only a rotary, but also a rising and falling movement, and further for water circulation through the hollow shaft and the stirring arms carried by it, while the blast is admitted at the top of the ash pit, and directly into the hot zone. According to this Specification, it is proposed

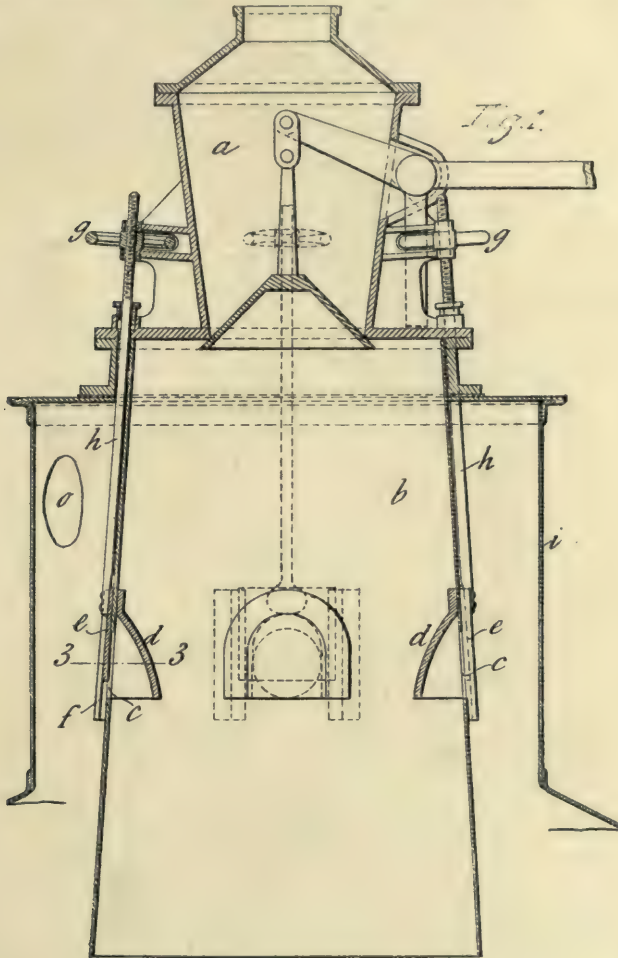


FIG. 73.—MOND GAS PRODUCER, BRITISH PATENT NO. 28,031 OF 1903.

to combine with the general form of producer shell and grate described in the first mentioned specification the stirring arms and central blast as described in Specification 10,049 of 1900, and to provide separate blast inlets for the central blast and for the circular grate provided with regulating valves, so that the air or mixture of air and steam used for burning the fuel in the producer is made to enter partly through the grate

and partly through the central blast in such proportions as may be found useful to obtain the best quality of gas and the largest yield of by-products. If desired stirring arms may be placed in the bell as well as in the main body of the producer in order to obtain a uniform stirring and breaking up of any caked material. The construction indicated

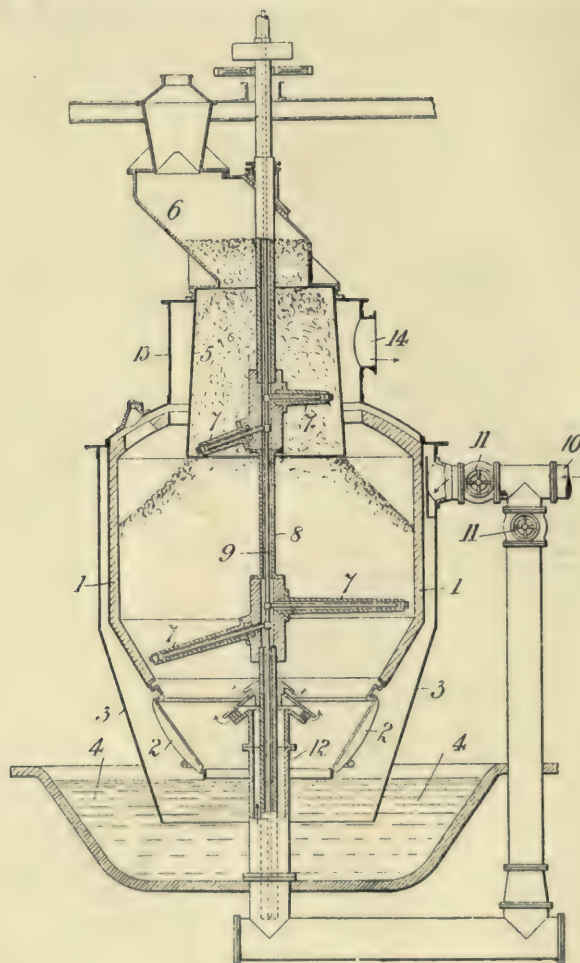


FIG. 74.—MOND GAS PRODUCER, BRITISH PATENT NO. 6719 OF 1904.

is stated to be particularly useful in connection with ammonia recovery plants.

Fig. 74 represents in sectional elevation one form of a producer constructed in accordance with this Specification. 1 is the lining of the producer, terminating at its lower end in the grate 2, while 3 is the surrounding jacket dipping into the water seal 4. The bell 5 extends below the top of the lining 1, and is connected to a suitable charging

chute 6. The stirring arms 7 are carried on a hollow shaft 8 provided with water circulation through 9, and mounted as described in Specification 10,049 of 1900. The air blast inlet 10 is branched and provided with valves 11, by which the amount of air passing through casing 3 to the grate bars, and that passing to the interior of the fuel through blast box 12 can be regulated at will depending upon the result desired. The gases generated rise up into casing 13 surrounding a part of bell 5, and pass out through discharge 14.

Fig. 75 shows the most recent design of Mond producer for by-product recovery, and Fig. 76 the non-recovery type. Measuring the efficiency of a producer by the ratio of heat units in the gas produced to the heat units contained in the fuel gasified, the following features in design and construction are claimed by the makers:—(1) Adequate but not excessive grate area, giving a uniform and effective distribution of the blast to the fuel bed, the angle of incidence of the blast being adjusted to suit the diameter of the producer. (2) A deep bed of incandescent fuel of sufficient thickness to ensure a maximum reduction of the CO_2 to CO , and the complete decomposition of the steam. (3) A layer of ashes supporting the incandescent fuel and resting on the bottom of the producer lute (not on the grate bars), thus preventing the escape of unburnt carbon into the water-seal. (4) Introduction of the steam-saturated blast at a point where it comes into immediate contact with the incandescent fuel zone, without previously passing through the ashes which tend to obstruct its passage—a practice which in some water-sealed producers causes cooling and condensation of the steam in the blast, and consequent deterioration in the quality of the gas. (5) Accessibility to the ash zone for the purpose, with clinkering fuels, of removing clinker. (6) Facilities for evenly removing ashes from the water-sealed bottom. (7) Absolute continuity of operation and uniformity of quality and quantity of gas produced during the cleaning of the fire. (8) Provision for the proper distribution of the fuel in the producer in order that the fuel bed may be maintained at an even thickness, thus avoiding blow holes, etc. (9) Disposal of the grate in such a manner that the greatest possible depth of fuel bed may be obtained without unduly increasing the over-all height of the producer. (10) Provision for the high super-heating of the steam-saturated blast and the cooling of the lining of the producer round the hot incandescent zone. This is of the greatest importance in by-product recovery plant for high temperature furnace work where gas of the highest heating value is required, along with the simultaneous recovery of the maximum amount of sulphate of ammonia. It is also of marked advantage in dealing with fuels of a highly caking tendency, and has resulted in the

successful gasification of refractory fuels which could not previously be worked to advantage.

The above features are claimed to result in the attainment of a "hot gas" efficiency of up to 85% of the total heat energy contained in the fuel.

Ammonia recovery is not usually undertaken in Mond plants

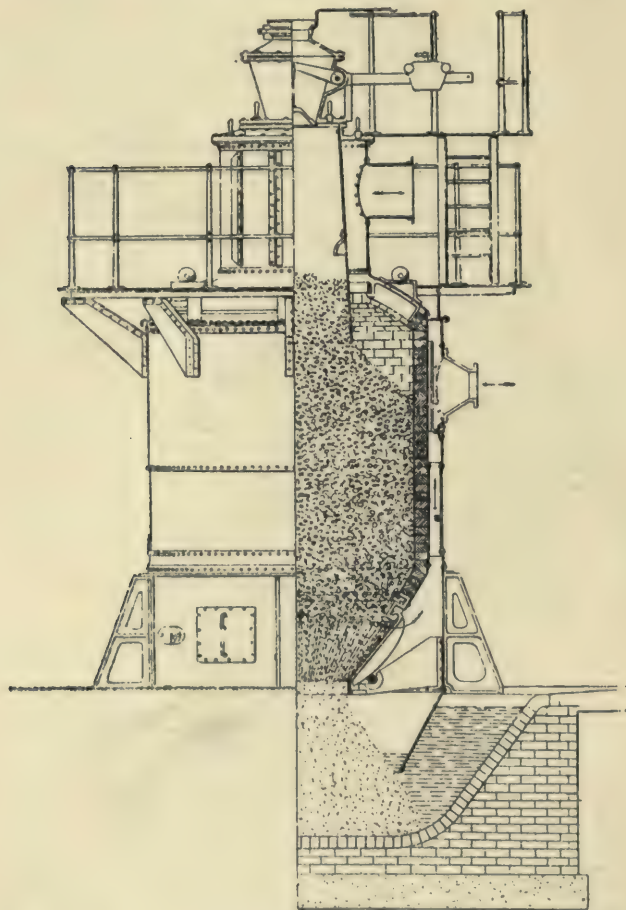


FIG. 75.—MOND PRODUCER FOR BY-PRODUCT RECOVERY PLANT.

where the fuel used is under one ton per hour. The percentage recovery is frequently 70% to 75% of the total nitrogen contained in the fuel, or about 100 lbs of sulphate of ammonia per ton of coal gasified, assuming coal to contain 1.4% of nitrogen.

For every ton of coal gasified two and a half tons of steam are

required, though of this apparently large quantity only one and a half tons have to be supplied from outside sources, as about one ton of steam is continually recovered by cooling the gas in specially designed heat interchangers. Exhaust steam at, or above, 2 lbs. pressure can be used for saturating the blast, but the supply must be constant and not intermittent. In power plants the whole of the steam required can be

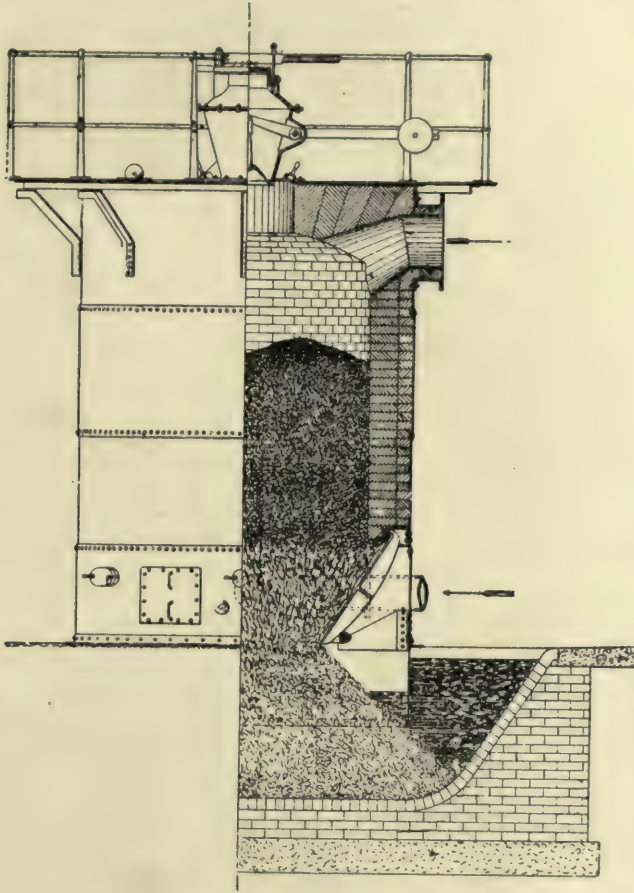


FIG. 76.—MOND PRODUCER FOR NON-RECOVERY PLANT.

raised, in specially designed boilers, from the hot waste gases from the gas engine exhaust.

A highly saturated air blast is essential to the recovery of sulphate of ammonia, as it keeps down the temperature of the producer, and thus prevents the destruction of the ammonia formed by the combination of the nascent hydrogen (derived from the steam) with the nitrogen in the fuel. Incidentally, the low temperature maintained

allows of the gasification of some fuels which would be unworkable in any other type of producer on account of caking and clinkering tendencies.

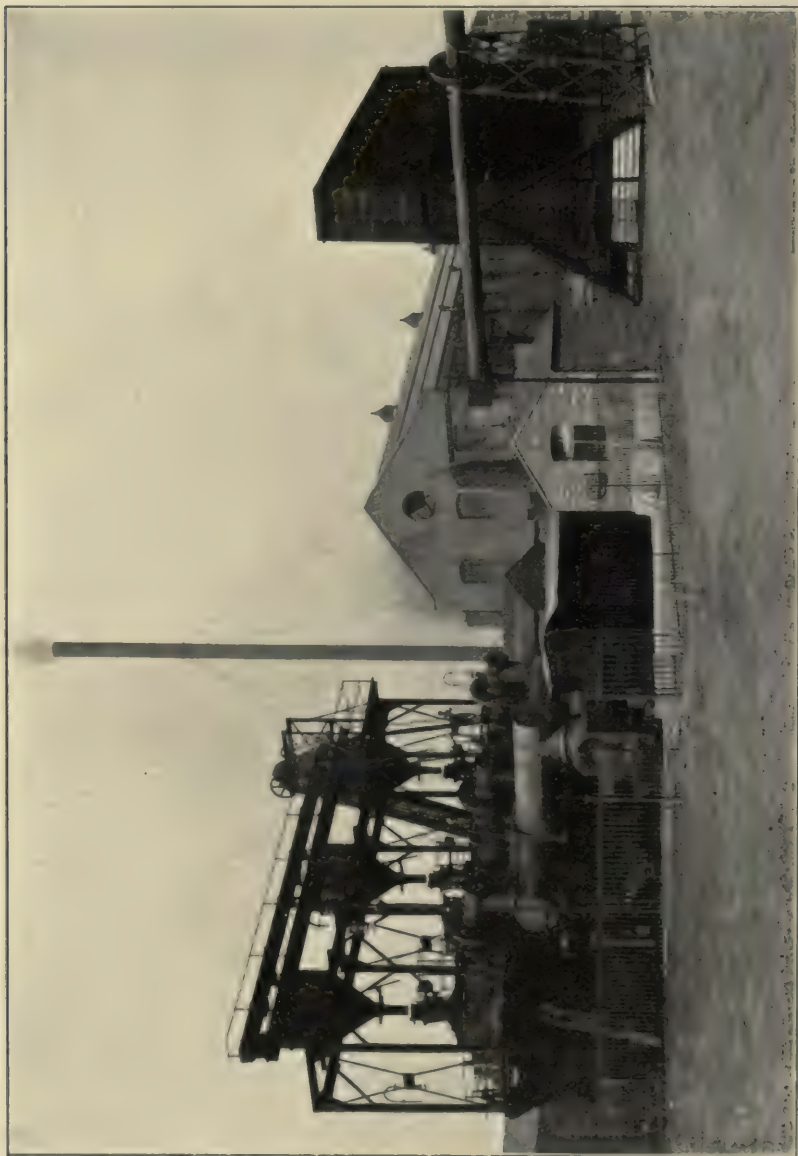


FIG. 77.—6,000-H.P. MOND GAS PLANT FOR POWER AND HEATING PURPOSES AT MESSRS. CAMMELL, LAIRD & CO.'S TRANMERE BAY SHIPYARD, BIRKENHEAD.

Coal is delivered direct from tip wagons into the elevator boot, from whence it is raised and distributed to overhead storage bunkers, from which it is charged into the producers by means of suitably de-

signed charging hoppers. The producers are provided with water seals, and are constructed to allow the ash, which is the only residue, to descend to the water, from which it is easily removed during working with-

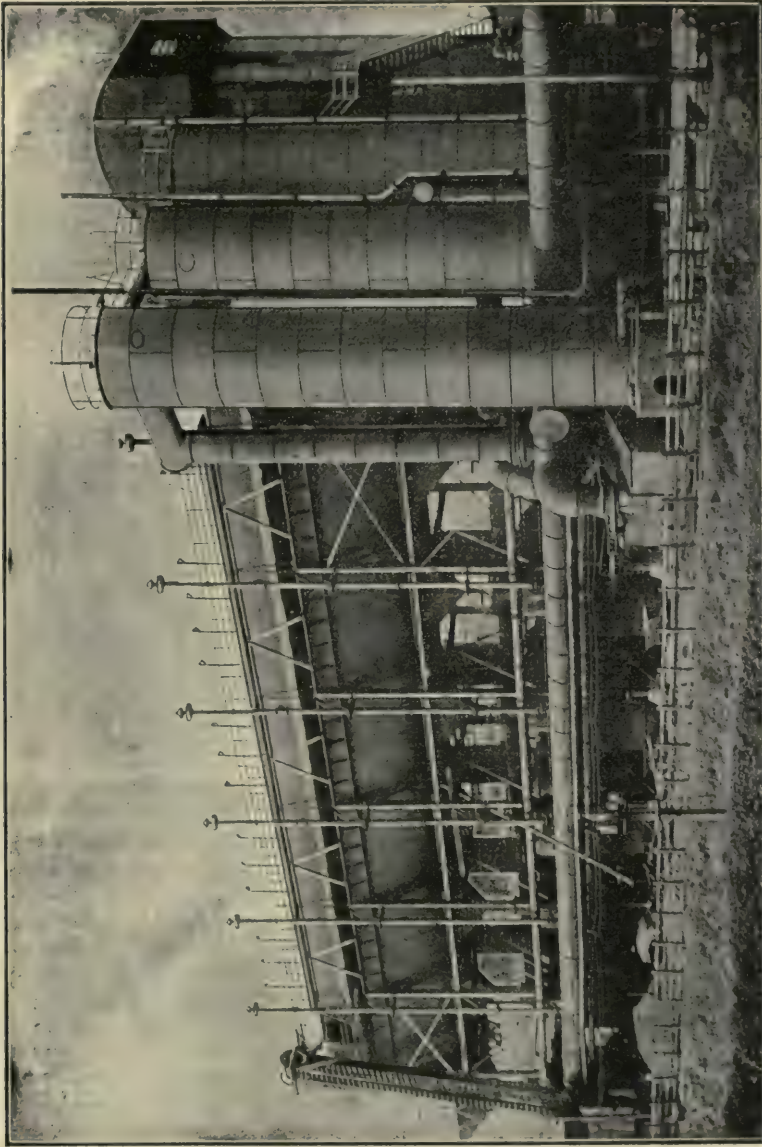


FIG. 78.—MOND GAS PRODUCER INSTALLATION, BY THE POWER GAS CORPORATION, LTD.

out interfering in any way with the quantity or quality of the gas made.

On leaving the producer the gas passes through a tubular superheater or heat interchanger, so constructed that part of the heat of the

gas entering it is transferred to the blast of saturated air on its way to the producers. By this means the producers are fed with a moist and highly superheated air blast, which ensures the uniform production of

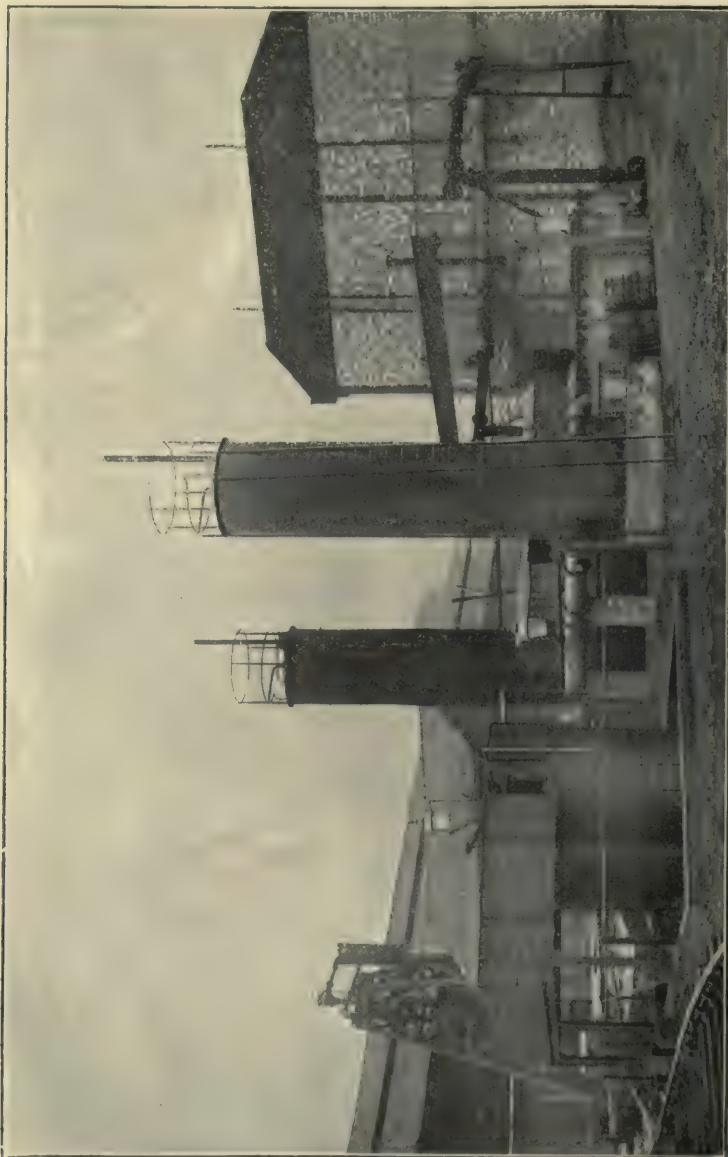


FIG. 79.—MOND GAS PRODUCER INSTALLATION, BY THE POWER GAS CORPORATION, LTD.

a gas of high heating value—about 140 to 150 B.T.U. per cubic foot, nett value.

Leaving the superheater the gas enters the mechanical washer, a

rectangular chamber, where it is thoroughly washed with water thrown up into a fine spray by a system of rapidly revolving dashers. In this washer the gas is freed from all dirt and a large part of its tar, while its temperature is further reduced. The dust and tar are removed by means of water-sealed cleaning lutes.

From the mechanical washer the gas is led to the ammonia absorber (which may be of a horizontal or vertical type) where it meets in very intimate contact a spray of weak sulphuric acid, which fixes the ammonia, forming a solution of sulphate of ammonia, which is withdrawn automatically and either sold in the form of "gas liquor" for re-distillation, or further concentrated by evaporation in special vacuum boiling-down pans until supersaturation is reached, when, on cooling, the solid sulphate of ammonia crystallises out, and is dried in a centrifugal machine and bagged for market.

Relieved of its ammonia the gas now enters the first gas cooler, where it is subjected to cooling and cleansing by means of intimate admixture with a finely-divided spray of cold water. Further cooling and cleansing takes place in a second gas cooling apparatus, traces of light tar being deposited during the cooling and removed by means of water-sealed lutes, or settling tanks. Finally, the gas leaves the plant at a temperature of about 30° C. (containing about 4% water vapour) ready for use in furnaces. If required for power purposes it is subjected to further mechanical washing and, finally, to dry scrubbing, to remove the last traces of tar before it reaches the gas engine valves.

The blast for the producers is supplied by means of positive blowers. On its way to the producers the air is forced through an air heater, where it comes into contact with a fine spray of hot water. This hot water is obtained from the gas cooler, and it will thus be seen that the heat taken from the gas is given to the air, water acting as the heat conveying agent. The hot water from the gas cooler having given up its heat to the air in the air heater, is returned cold to the gas cooler, and by this ingenious arrangement a continuous cyclical exchange of heat takes place, and a large proportion of the steam required to saturate the blast is recovered.

The gas coolers and air heaters are designed to suit local circumstances, and may be of either horizontal or vertical type.

The Kerpely Gas Producer.—The Kerpely producer, of which the makers are Messrs. E. G. Appleby and Co., of 10, Victoria Street, London, S.W., embodies the principle of the revolving grate, together with a special water trough, ash scraper, and duplex air and steam supply. Fig. 80 is a section of the producer, Fig. 81 shows an installa-

tion of nine Kerpely producers, the view being along the charging stage, and Fig. 82 shows the ash pan and drawing stage.

The use of a water trough in connection with gas producers having fixed seal aprons has been largely adopted by makers of fixed-grate producers. It is claimed that in the Kerpely producer was first realised the advantage of making the water trough rotate on a central axis; for this purpose rollers under the outside path of the water trough were originally used, but these were discarded in favour of balls running in a ball race.

The advantage of making the water trough rotate is that by this movement the ash from the producers is brought up against a scraper, which automatically discharges the ash over the side of the water trough, and it is the combination of these two features, the rotating water trough and the ash scraper, that is one of the essential details of the patents relating to the Kerpely producer.

The ash scraper is constructed to suit the shape of the ash trough, and is placed at a convenient angle for the most efficient discharge of ash. The action of the scraper is not a horizontal one, which only pushes the ashes aside without piling them up. Kerpely claims to have originated the piling up of ashes vertically, consequent upon the special construction of the rotating ash trough.

Following up these two features, and in combination with them, a grate of special construction has been devised, which, being placed eccentrically upon the rotating water trough, rotates with it, and ensures (a) the automatic discharge of ash and clinker into the water trough, as well as (b) the perfect control of the fuel bed.

The revolving mechanical grate consists of a single oblong and spherically shaped cone fixed eccentrically upon the water trough, and provided with a nearly flat oblong shaped top. This cone comprises a number of massive iron plates, of which the grate is built up in sections. Through the spaces between these grate plates, which cannot choke up with ashes, the air and steam is dispersed over the whole area of the fuel bed, ensuring the maximum gasification capacity per square foot of grate area. The grate has a continuous and not an intermittent action in crushing any formation of clinker that may exist. The clinker, when entering the ash zone, is caught by the numerous edges of the grate, and pressed horizontally sideways against the seal apron, and consequently broken or crushed into small fragments, which, dropping into the water trough, are removed by the ash scraper.

The water-cooled jacket has been proved by experience to be essential for the gasification of clinkering and caking fuels. In the Kerpely producer water is kept continually circulating through the

jacket, and the makers state that no clinker can adhere to the water-cooled surfaces; the clinker in the producer is automatically disinte-

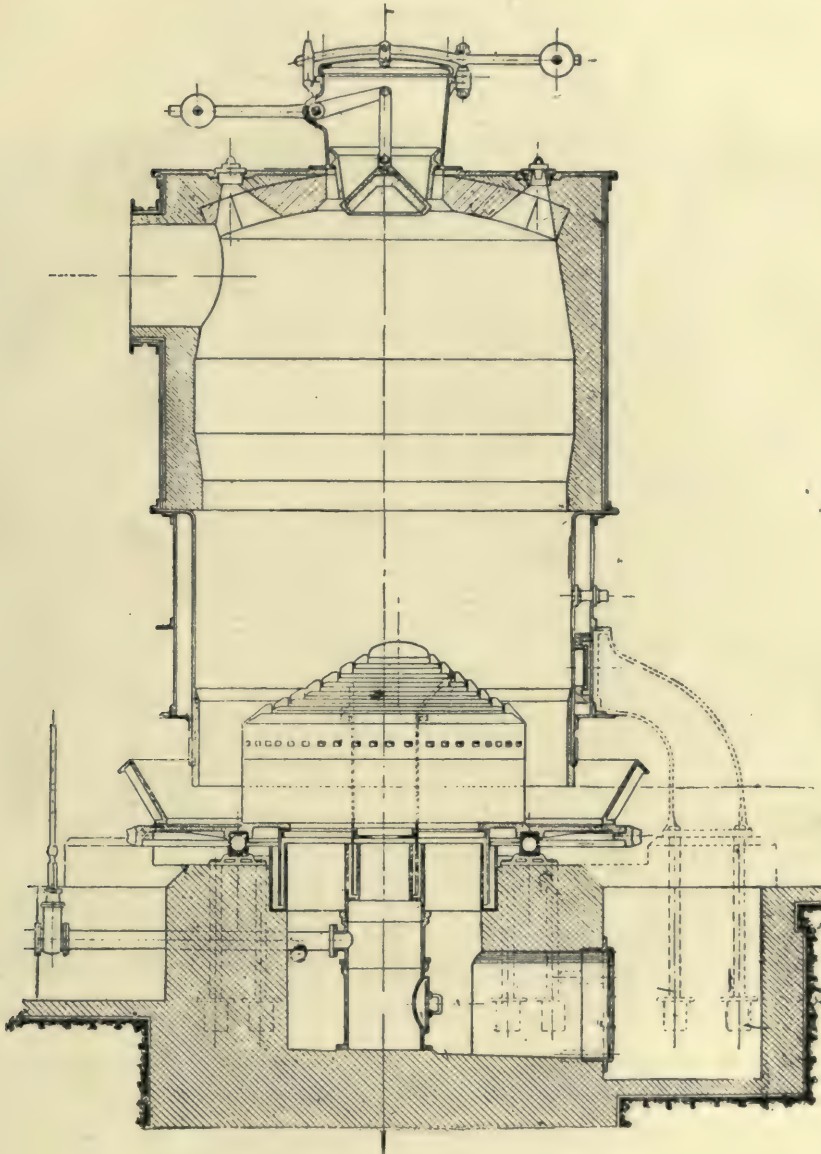


FIG. 80.—KERPELY GAS PRODUCER: SECTION.

grated by the rotating action of the grate against the water-cooled plates. The water-cooled jacket obviates the excessive poking re-

quired to break down the clinker which adheres to brick-lined producers, and also the use of excessive steam.

The ash and clinker accumulating in the bottom of the producer are automatically discharged by coming up against the ash scraper, and by regulating the position of this scraper the ash discharge is adjusted to suit the fuel, the grate being in continuous rotation in order



FIG. 81.—KERPELY GAS PRODUCERS: VIEW ALONG CHARGING STAGE.

to loosen the fuel bed. The ash scraper is constructed to suit the shape of the ash trough, and consequently the ash is not only pushed aside, but is piled up and discharged into the ash shoot. The ash scraper ensures an automatic discharge of the ash and clinker from the bottom of the producer, and also its continuous working.

The supply of air and steam to the producer is separately conveyed to the inner and outer sections of the grate, and being transmitted to the fuel bed through the spaces between the plates of the

revolving grate, ensures uniform combustion over the whole grate area; thus when the producer works too hot at the edge and is too dark in the middle, the outer air pressure is reduced and the inner air pressure raised until an even temperature is produced all over the surface of the fuel bed and *vice versa*. The duplex air and steam supply ensures uniform combustion in the fuel bed.

At the Leeds meeting of the Institution of Mining Engineers on

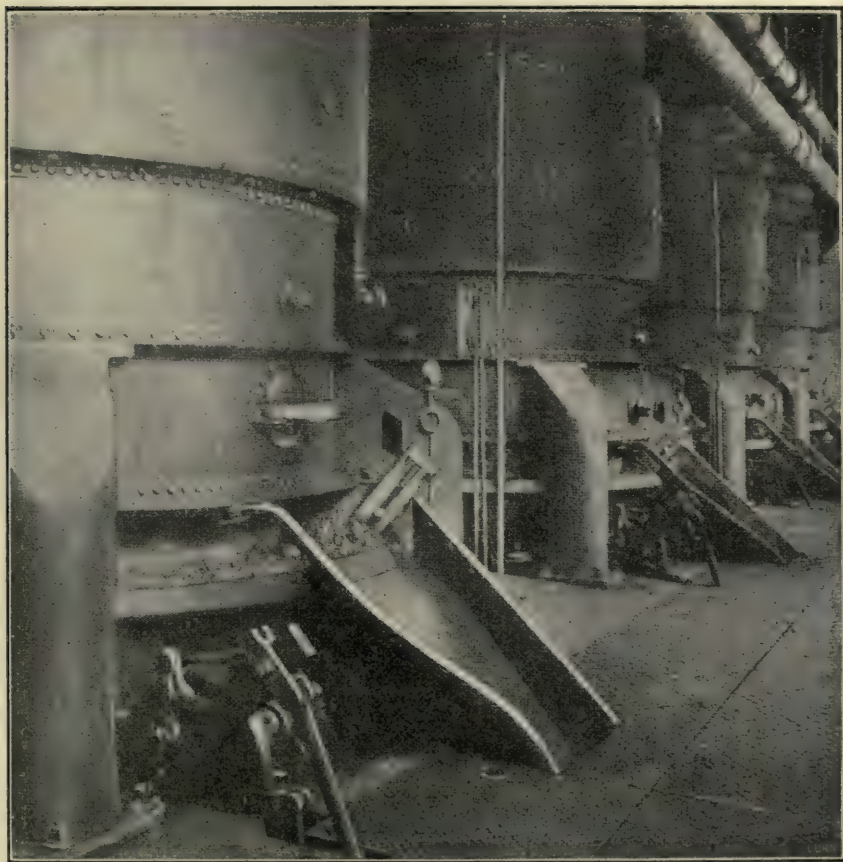


FIG. 82.—KERPELEY GAS PRODUCERS: SHOWING THE ASH PAN AND DRAWING STAGE.

September 15th, 1915, in the course of a paper on "Colliery Power and By-Products from Unsaleable Fuel," Mr. Mansfeldt H. Mills gave descriptions (see abstract report in *Gas World*, October 2nd, 1915) of an installation of seven Kerpely producers at the works of the Shelton Iron, Steel and Coal Company, and of three at Littleton Colliery. The former were said to gasify effectively a low-grade fuel, locally known as "batts," which had previously been thrown away, and consisting of

interstratified coal and shale picked from the colliery screening belts. During the coal strike of 1912, the Company kept two Siemens furnaces entirely working on gas from this low-grade fuel, but in general practice for steel smelting a good fuel was mixed in about equal proportions; nevertheless, about 11,000 tons of "batts" had been used in a year. At Littleton the producers were of the simplest type, having no firebars or grate. This plant is operated without any cleaning fan, or mechanically driven apparatus, except a small Roots blower for the air and steam blast. As the washer is worked entirely on tar as a cleansing medium, no added water for cooling or cleaning purposes is brought into contact with the gas. The result of this is that the gas is subjected to a condensing operation, no extra water having to be evaporated, and as a consequence the tar effluent contains the minimum percentage of water. The separation of tar and water is also naturally effected in the smallest compass. The cost of erecting such a plant was stated to be £2 10s. per brake-horse-power, including cooling and cleaning plant, as also the blowers and engines, steam boiler, and gasholder, but not including excavations and builder's work. The gas is used for driving two large gas engines, which generate electric power for running all the machinery, both above and below ground, excepting the winding engines. Some "average" results were given, obtained in Kerpely producers, from a low-grade fuel analyzing as follows:—Carbon, 58.30%; hydrogen, 3.70%; nitrogen, 1.10%; oxygen, 8.06%; moisture, 6.90%; ash, 21.10%; sulphur, 0.84%. A producer 10 feet in diameter, the author said, will gasify an average of 24 tons of this class of coal per twenty-four hours; about 7 cwts. of coal are fed into the producer every twenty minutes, the pokers being used every half-hour. About 0.25 lb. of steam per lb. of fuel is used when by-products are not recovered, and quite 25 lbs of steam per lb. of fuel when sulphate of ammonia is recovered. The water gauge at the air-blast inlet generally shows about 4 inches, but when ammonia is recovered the water gauge will run up to quite 18 inches, this depending upon the condition of the fuel. The air is saturated with steam at 45° C., which must be increased to 95° C when ammonia is recovered. The following is an average analysis of the gas from such a fuel as that named, with the producer working in good order and under non-recovery conditions:—Carbon dioxide, 4.0%; carbon monoxide, 26.5%; hydrogen, 10.5%; methane, 3.0%.

The temperature of the gas leaving the producer is about 500° C. An average of 150,000 cubic feet of gas is obtained from each ton of this fuel.

The Moore By-Product Recovery Gas Producer.—The Moore gas producer is shown in plan, elevation, and cross-section in Plate X. In describing this producer, the *Journal of Gas Lighting* recently observed that the most important factor in the economy of by-product recovery producer practice in those types of plant which are best known at present is that of steam consumption, which amounts to about 1.5 lbs. per pound of coal gasified. This factor, taken in conjunction with the high initial cost of the plant, including the extra capital expenditure on steam boilers, the low thermal efficiency of the plant, and the wetness of the gas, frequently acts as a deterrent to the installation of by-product recovery gas producers. The Moore producer is claimed to be an entirely new departure, and is designed with the object of gasifying the fuel under conditions which permit of the recovery of the tar and a large amount of the available nitrogen as ammonia without sacrificing the thermal efficiency of the plant or the quality of the gas. It cools the gases rapidly by means of a water-jacket, and is stated to permit of the use of a much smaller proportion of steam in the blast without any sacrifice of the ammonia. The following particulars are extracted from a very full description in the *Engineer*.

The producer shaft, which is made partly of $\frac{3}{8}$ -inch steel plate and partly of brickwork, is conical in vertical section, and ovoid in plan. There are three distinct sections: The top section is air-cooled, about 3 feet deep, and extends from the underside of the producer top plate to the top of the water-jacket. In this section the gas outlet pipe is arranged. The middle section is water-cooled, being surrounded by a water-jacket about 6 feet deep. The water-jacket outer casing is composed of $\frac{3}{8}$ -inch mild steel plates, and the casing is furnished with all the necessary boiler fittings. The pressure in the jacket is practically atmospheric, and therefore water can be fed into it by means of a Royles feed regulator, thus maintaining a constant level of water. The bottom section is composed of a mild steel casing, which contains the firebrick portion of the producer and also the grate. The former is lined with special fire-bricks designed to suit the shape of the producer. In this section, which is about 4 feet deep, the highest temperatures in the producer are to be found, and the actual gasification of the coked fuel takes place there.

The grate is about 3 feet deep, and is composed of eight or more cast-iron sections in order to simplify manufacture. These sections are made so that a bridge is formed across the producer on the narrow axis, thus dividing the grate into two inverted cones. The

grate as a whole is supported on cast-iron brackets and section plates, which are fixed to the shell of the bottom section. The plates also support the brickwork of the producer. The ash-hopper, which also has the shape of an inverted cone with flattened sides, is fixed to the angle or flange on the lower edge of the bottom section casing. This hopper is made of mild steel plates or cast-iron, and is arranged with sides sloping at an angle sufficient to allow the ash to travel down easily. Poker-holes are formed in it, and these can be used, if necessary, to facilitate bringing down the ash. In cases where one unit only is installed a drum-cock valve is fitted in addition to the shutter-valve, to avoid any drop in pressure in the producer when the ash is being removed.

Supported on cast-iron brackets fitted to the side of the ash-hopper are two circular cast-iron ash-supporting tables, fixed on the same axis as the inverted cones forming the grate. These tables are arranged below the bottom of the grate, and are of such dimensions that they overlay the bottom circle of the grate cones by a larger amount than that necessary to accommodate the angle of repose of the ash. Two steel shafts, each carried in a bearing on the lower portion of the ash-hopper shell, and placed immediately below the centre of each ash-table, pass upwards and through a hole in the centre of the table. On each shaft is fixed a cast-steel arm, which rotates above and just clear of the table. At the lower end of each shaft a ratchet-wheel and pawl are fixed, thus allowing the shaft and arm to be rotated by hand or by mechanical means, and making it possible to bring down the ash from any particular portion of the producer as required.

The two inverted cones constituting the grates are perforated with vertical slots, which form admission ports for the blast to the fire. These slots are arranged round the outer circumference of the cones. The blast-box surrounding the grate is formed by the lower portion of the bottom shell, and provision is made for easy access to it for inspection at any time. The blast comes into this box by two inlet-pipes, each of which enters on the same centre line as the grate circles. The pipes are each provided with a screw-down valve close up to the producer shell, so that the supply of air to either side of the blast-box can be regulated. Immediately below the grate, in the walls of the ash-hopper, poker-holes are provided to allow access to the ash on the tables, should it be necessary to remove pieces of clinker. To assure a constant and well-distributed feed of coal, the producer is provided with a fuel container, into which the fuel is charged through a hopper fitted with a gas-tight bell valve and cover.

The cleaning and cooling plant is claimed to be simple and com-

Fig. 1

170 a





The cleaning and cooling plant is claimed to be simple and compact, and to require no skilled attention; and as the recovery portion of the plant works automatically, labour charges are reduced to a minimum. The outlet and downcomer pipe from the producer is arranged with the smallest possible number of horizontal lengths, so that the dust will have the minimum amount of length on which to deposit; otherwise it slopes or drops vertically into the dust-lute. This is a mild-steel box provided with a water-seal through which the dust can be removed when required.

The inlet-pipe is on the side nearest the water-seal, and is connected to a cone-shaped pipe inside the box. This pipe is provided with slots so as to break up the volume of gas into a number of streams. A plate divides the box, and is provided with slots (similar to those in the cone) for further cleaning purposes. Provision is made for cleaning these slots through handholes in the box. The exit-pipe from the dust-lute leads to an omnibus pipe, which distributes the gas to the atmospheric condensers or coolers. These are of the ordinary type, and consist of four batteries of 4-inch cast-iron pipes fitted into cast-iron chests. Between the second and third chests is placed a Pelouze and Audouin tar-extractor, the inlet of which is connected by a gas-collecting pipe to the second chest, while the exit is joined by a gas-distributing pipe to the third chest. The fourth chest from the producer is connected to a collecting pipe which leads the gas into the main communicating with the ammonia scrubbers.

The scrubbers are made of $\frac{3}{8}$ -inch and $\frac{1}{4}$ -inch mild steel plate, and are 35 feet high, the diameter varying according to the size of the plant. They are filled with wooden grids to allow of a large scrubbing surface. The lower portion of each tower is usually arranged as a liquor-tank, from which the liquor is circulated by small centrifugal pumps. The first scrubber is provided with pipes at the top for distributing the liquor over the scrubbing surface; the second with two sets of distributing pipes—one at the top for distributing clean water, and the other half-way down for distributing weak liquor. By this means the gas is efficiently scrubbed free from ammonia before it leaves the plant. For small plants a tumbler, provided with a mechanical counter, is arranged on the top of the second tower, to allow the clean water added to be measured, and within certain limits the strength of the strong liquor leaving the first tower can be regulated. For larger plants a third tower so fitted is used. Between the first and second towers is an overflow and circulating box, which allows the weak liquor to pass from the second to the first tower, and the strong liquor to

overflow from the first tower to a storage tank. Tanks are provided for tar and ammoniacal liquor, and they can be placed under or above ground as required. The chests of the atmospheric condensers are connected by circulating pipes, which allow the tar and condensed steam containing ammonia to flow into the chest nearest to the producer. The auxiliary machinery required for the plant consists of an air blower and three belt-driven centrifugal pumps, which in small plants are worked from the blower engine.

The following particulars will give an indication of the working of the plant:—The fuel is charged through the hopper into the container, from which it descends, and meeting the hot gases has its moisture driven off. In the water-jacketed zone the hot ascending gas and vapour are immediately cooled to a temperature below that at which ammonia is decomposed. The fuel is here also destructively distilled at a low temperature, so that tar and ammonia are formed, and the coke is rendered more porous, and more suitable for gasification in the combustion zone. The resistance offered to the blast by the fuel in the producer is low. This is partly accounted for by the easy slope of the producer itself, but is chiefly due to the simple design of the recovery plant. A very low pressure at the outlet from the producer is sufficient to force the gas through the rest of the plant, and incidentally this greatly reduces the loss of gas due to poking, etc.

The gas leaves the producer at a temperature of about 180° to 230° C., and contains, it is stated, a relatively small amount of steam and dust. After the dust has been removed, the cooling of the gas is effected by passing it through the first series of atmospheric condensers, where it is cooled to a temperature suitable for extracting the tar. Thereafter the gas is passed into the second series of atmospheric coolers, where it is cooled to the outside temperature. The gas is then led into the scrubbing-towers, through which the weak and cooled liquor from the coolers and clean water circulate, and extract the ammonia, yielding liquor containing from 1% by weight of ammonia. This liquor is claimed as suitable for manufacturing ammoniacal liquor and ammonia salts.

The tar from the Pelouze and Audouin extractor runs into the second chest, and flowing, along with the water, to the first chest, where it meets the hot gas entering the condenser, is heated and freed from the finely divided water. The tar and virgin liquor flow from the first chest into a small tank, where they are separated by gravity. The tar flows into a storage well, and the liquor is run through a small heat-

interchanger into the second tower, and used for scrubbing the gas. For the manufacture of sulphate of ammonia, the liquor is pumped through an ammonia still of any standard type, lime being added to liberate the ammonia present as fixed salts. The steam and gases are passed through a saturator, where the ammonia, coming in contact with sulphuric acid, forms sulphate of ammonia, which can be removed by ejector as required. The foul gas and steam pass on, and are mixed with the producer blast. By this means the amount of steam required is reduced to the minimum, and the trouble of purifying the foul gas from the sulphate plant is eliminated.

The gas leaving the producer plant is claimed to be clean and practically free from suspended moisture, and to be eminently suitable for boiler firing and furnace work. Before being used in a gas engine, it is desirable to further purify it by passing it through a sawdust scrubber. As to the dryness, tests taken give the following average results:—

| | |
|--|-------------|
| Atmospheric temperature (in shade) | 68° Fahr. |
| Temperature of gas leaving plant | 70° Fahr. |
| Suspended moisture per 100 cubic feet of gas | 910 grains. |

Equal to 10.2 per cent. on saturation vapour.

The dry bottom grate is claimed to be a distinctive feature of the producer; and owing to the distribution of the blast the gasification of the coal is said to be perfect—the amount of carbon remaining in the ash not exceeding 1% of that calculated to be in the fuel. The steam required for the saturation of the blast does not exceed 1 lb. per pound of coal. Of this the water-jacket of the producer provides 50%, so that the requirements from outside sources do not exceed 0.5 lb.

The following figures, taken from the official trials of a plant recently installed, show the efficiency of the producer, and include the amount of coal used at the boiler for raising the extra steam required for the saturation of the blast:—

| | | |
|---------------------------------|--------------|--------------|
| Steam used in blast per pound | | |
| of coal gasified | 0.8 lb. | 1 lb. |
| | B.Th.U. | B.Th.U. |
| 1 lb. of coal into producer ... | 11,145 | 11,122 |
| Coal fired at boiler per pound | | |
| of coal gasified | 0.04 lb.=446 | 0.07 lb.=779 |
| | <hr/> | <hr/> |
| | 11,591 | 11,901 |
| Heating value of gas from 1 lb. | | |
| of coal net | 9,240.8 | 8,909 |
| Efficiency of plant | 79.7% | 74.8% |

The net available heat of the gas is claimed to be 25% greater than that obtained from ordinary recovery plants, and the saving of steam when gasifying one ton of coal per hour equal to 24 tons per day of 24 hours. The nitrogen recovered as ammonia when using 1 lb. of steam per pound of coal gasified is equal to that recovered in ordinary practice—viz., 80 to 90 lbs. of sulphate per ton of coal.

The following results of official tests allow a comparison of the effect of steam on the yield of sulphate of ammonia:—

| COAL. | | | | | 840 lbs. | 840 lbs. |
|---|-----|-----|-----|-----------|-----------|----------|
| Wet coal gasified per hour ... | | | | | 840 lbs. | 840 lbs. |
| APPROXIMATE ANALYSES. | | | | | | |
| Moisture | ... | ... | ... | 8.05% | 8.06% | |
| Volatile matter | ... | ... | ... | 30.45% | 30.42% | |
| Fixed carbon | ... | ... | ... | 49.35% | 49.21% | |
| Ash | ... | ... | ... | 12.15% | 12.31% | |
| | | | | | 100.00 | 100.00 |
| Nitrogen | ... | ... | ... | 1.274 | 1.288 | |
| BLAST. | | | | | | |
| Pressure | ... | ... | ... | 8.75 W.G. | 9.14 W.G. | |
| Saturation temperature | ... | ... | ... | 73.6° C. | 77.0° C. | |
| Total steam used per lb. of coal gasified | ... | ... | ... | 0.81 lb. | 1.0 lb. | |
| Steam raised in producer water jacket per lb. of coal gasified | ... | ... | ... | 0.5 lb. | 0.5 lb. | |
| Air used per lb. of coal in c. ft. at N.T.P. | ... | ... | ... | 28.8 | 28.3 | |
| SULPHATE OF AMMONIA. | | | | | | |
| Yield per ton of coal gasified... | ... | ... | ... | 67.3 lbs. | 90.5 lbs. | |
| Nitrogen recovered as ammonia | ... | ... | ... | 50.3% | 66.9% | |
| GAS. | | | | | | |
| Yield per ton of coal gasified in c. ft. at N.T.P., B.Th.U. net | ... | ... | ... | 120,000 | 123,500 | |
| Heating value per c. ft., B.Th.U. net | ... | ... | ... | 172.5 | 161.6 | |
| ANALYSES OF GAS AS SAMPLED. | | | | | | |
| CO ₂ | ... | ... | ... | 8.2% | 11.6% | |
| CO | ... | ... | ... | 22.2% | 18.4% | |
| CH ₄ | ... | ... | ... | 2.8% | 2.2% | |
| H ₂ | ... | ... | ... | 23.2% | 26.0% | |
| N ₂ | ... | ... | ... | 41.8% | 40.0% | |
| Water vapour | ... | ... | ... | 1.8% | 1.8% | |
| | | | | | 100.0 | 100.0 |

The tar produced contains 36% by weight of oils, and yields pitch of good quality.

The Duff Gas Producer.—Fig. 83 shows the construction of the producer of A. B. Duff, of Pittsburg, U.S.A., as described in his British Patent No. 16,164 of 1903, the illustration being reproduced from the Patent drawing. In this specification, Duff observed that “in gas producers as hitherto generally constructed the air (or air and steam) has either been distributed by means of gratings placed more or less

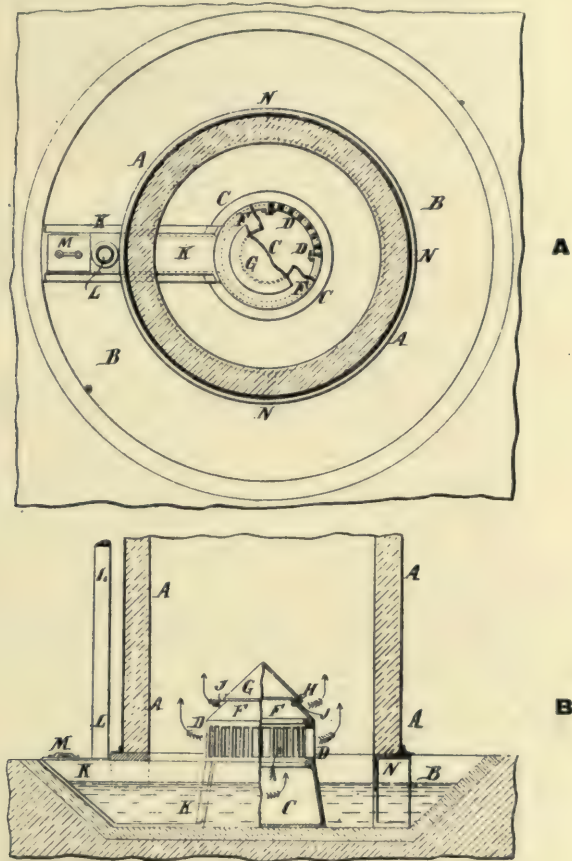


FIG. 83.—DUFF GAS PRODUCER, BRITISH PATENT NO. 16,164, OF 1903.

vertically in a bottom casing or by means of a louvred cone or hood delivering the air either downwardly or more or less horizontally. According to my present invention I combine these two methods of introducing the air (or air and steam) to the producer, superimposing the louvred cone on a casing having more or less vertical gratings. With this improved combined construction greater rapidity of action is secured, while the fuel is more perfectly reduced to ashes, as whilst the

combined blowers work simultaneously, the streams of air or air and steam from the two blowers at the same time act consecutively upon the fuel as the latter moves downwards, the upper conical blower delivering air to light up the fuel to incandescence, while the lower grating causes the air to act upon the fuel with a finer distribution, completely reducing the remaining partly gasified fuel to ashes."

Fig. A is a sectional plan, and Fig. B a sectional elevation of part of a producer with these improvements. The shell A of the producer, lined with firebricks, is circular in plan, the lower edges of the shell dipping as usual into the water in an ash trough B, the water forming a gas-tight seal, and the ashes or residues descending into the water seal being removed by means of rakes as usual. In the centre of the trough B there is built a casing C, upon which is superposed a ring of vertical gratings D, which, however, may be more or less inclined, and made up of sections bolted together. On the top of this ring there is placed a conical louvre ring F, and on the top of the ring F a cover G is secured by bolts and distance pieces H, at intervals, so that an air space J is left between the ring F and cover G, and the whole forms a louvred cone or hood having a closed top. Instead of there being only one louvre ring F, there may be a number, these rings being secured in position by bolts and distance pieces, so as to leave air spaces (J) between each, and being in all cases surmounted by a cover G. The air or air and steam for combustion or gasification of the fuel is supplied to the casing C, through a duct K, communicating outside the producer shell A with a blower pipe L. The air (or air and steam) passes from the casing C into the producer both through the gratings D and the air space (or spaces) J, and acts on the fuel in the improved manner hereinbefore described. The duct K is provided with a door M, through which a rake can be inserted to clean out any residues which may become deposited in the casing C. The duct K forms one of the supports for the producer shell A, other supports N, of the usual form, being also provided.

Fig. 84 is a reproduction of the drawing illustrating A. B. Duff's British Patent 16,243 of 1903, the object of which was "to provide an improved air-heating and steam superheating apparatus for gas producers which shall be simpler, stronger, cheaper, and more efficient than those hitherto in use, while the parts are arranged so that they are more accessible for examination or repair."

Fig. 84 is a sectional side elevation of the improved apparatus as applied to a producer. There is provided a vertical shell or casing A, of circular form, which is fitted with upper and lower tube plates A¹, A²,

and also with a transverse baffle plate A^3 , extending up from the lower tube plate to within a short distance of the upper tube plate. On each side of the baffle plate A^3 there are fixed between the tube plates A^1 , A^2 , two tubes B^1 B^2 , and C^1 , C^2 , the upper ends of which open into an upper space or box A^4 , formed above the upper tube plate A^1 , the lower ends of each pair of tubes opening respectively into inlet and out-

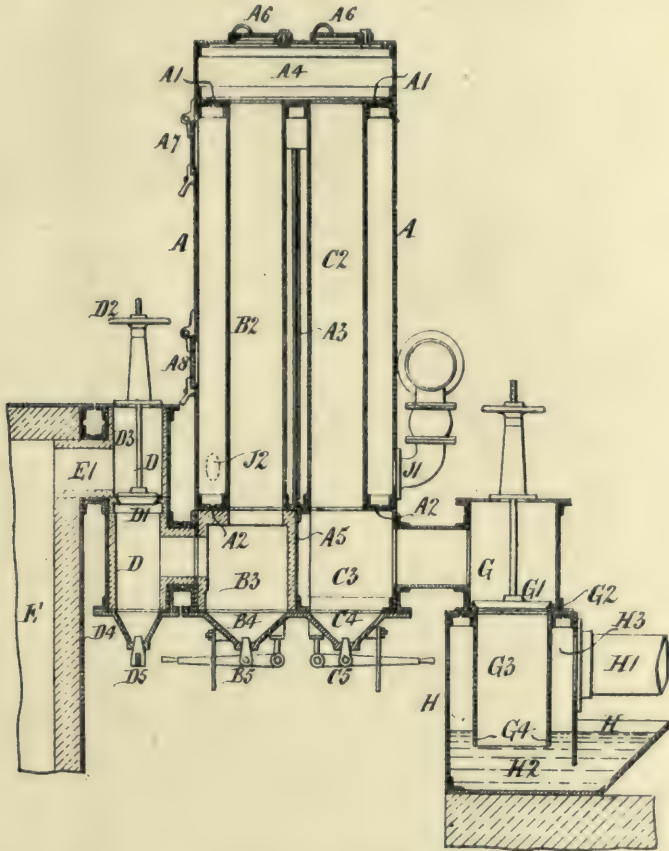


FIG. 84.—THE DUFF GAS PRODUCER, BRITISH PATENT NO. 16,243, OF 1903.

let bottom spaces B^3 , C^3 , arranged below the bottom tube plate A^2 , and formed by a cross partition plate A^5 . The inlet space B^3 is connected to a brick-lined chamber D , connected to the gas discharge outlet E^1 of the producer E , the chamber being fitted with a stop valve D^1 , operated by a wheel and spindle D^2 , D^3 . Similarly the outlet space C^3 is connected to an outlet chamber G , fitted with a stop-valve G^1 , and connected to a hydraulic lute washer or main H , fitted with a gas discharge pipe H^1 . The valve outlet G^2 opens into a downwardly extend-

ing casing part G^3 , which dips into the water H^2 in the washer, the bottom edge or end of such part being serrated, or it might be per-

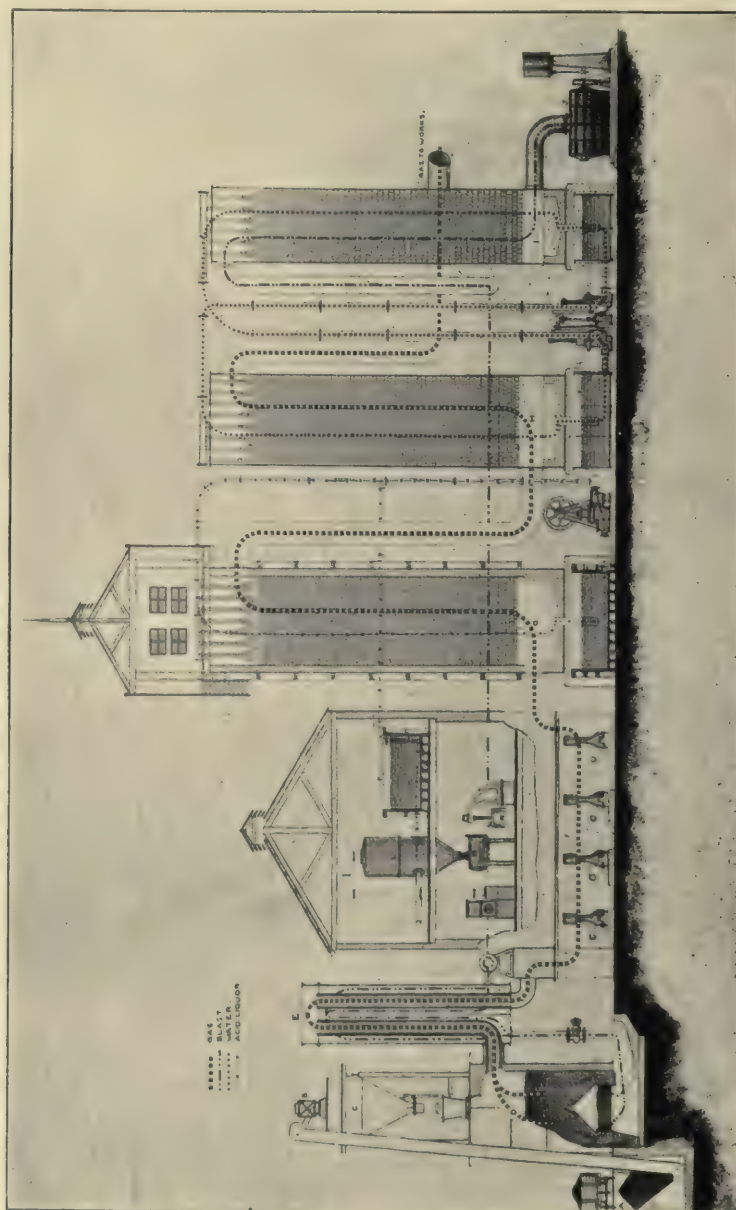


Fig. 85.—DIAGRAM SHOWING ARRANGEMENT OF A. B. DUFF PRODUCER PLANT.

forated with small holes. The lower ends of the inlet-valved chamber D , and the bottom spaces B^3 , C^3 , are provided with conically shaped

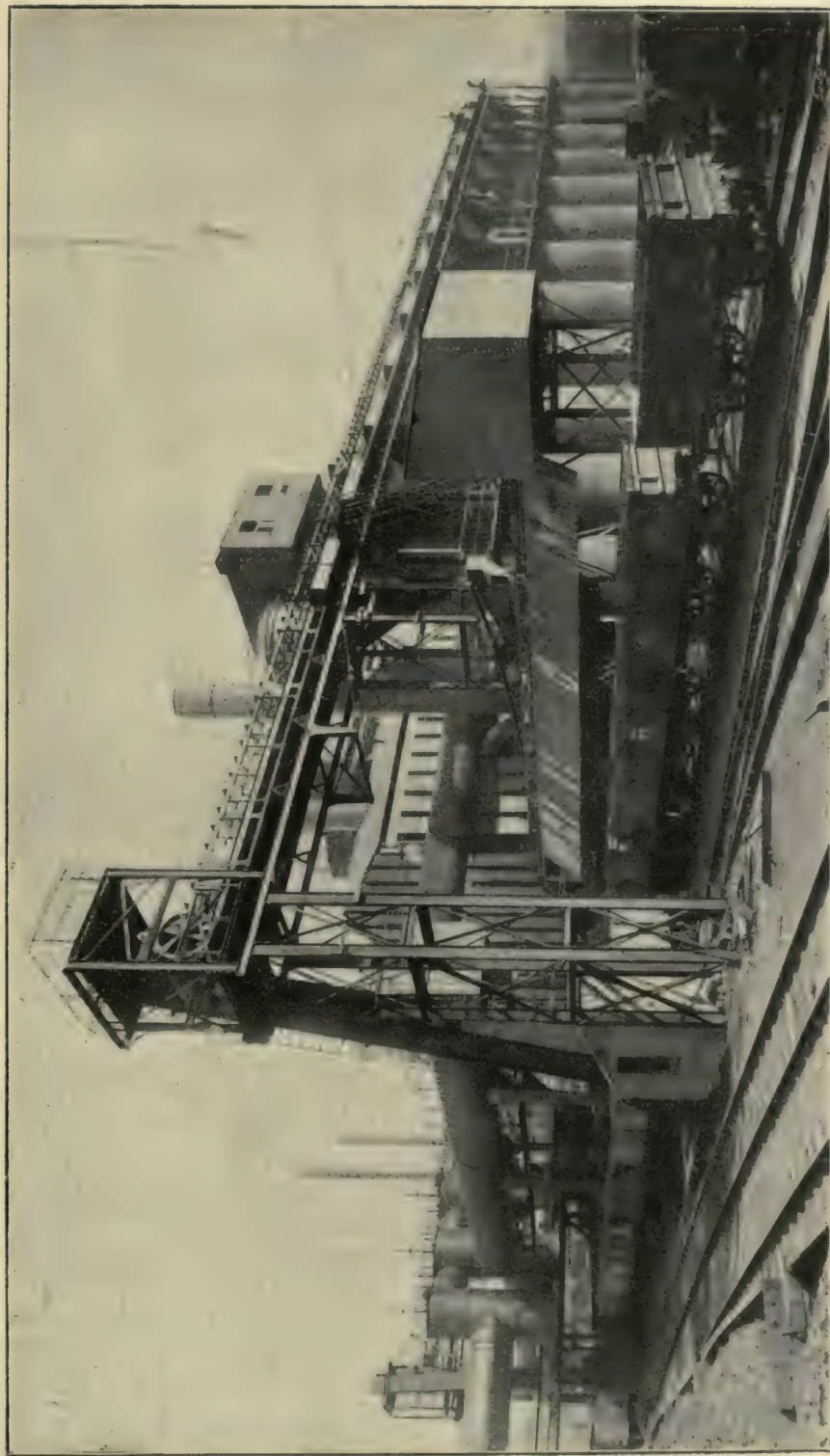


Fig. 86.—A. B. DUFF BY-PRODUCT PRODUCER PLANT BY THE GAS POWER AND I.V.-PRODUCTS CO.

dust-collecting boxes D^4 , B^4 , C^1 , fitted with handled plugs or stoppers, D^5 , B^5 , C^5 , while air inlet and outlet connections J^1 , J^2 are fitted to the circular shell A at a point a little above the lower tube plate A^2 , the outlet J^2 being connected to the blower casing or grate part of the producer E. A manhole door A^6 is fitted to the top of the circular casing A, opposite each vertical tube, and similar doors A^7 , A^8 are provided on the side of the casing, so that entrance can thus be conveniently obtained for examination or repair of the parts, ample room being left between the parts so as to allow a workman to get about with facility, and perform the necessary examination or repair.

The action of the apparatus is as follows:—The inlet and outlet gas valves D^1 , G^1 being opened, gas formed in the producer E passes out by the upper outlet E^1 into the inlet valved chamber D, and flows into the inlet bottom space, B^3 . From this space it flows upwards through the first pair of tubes B^1 , B^2 into the upper space or box A^4 . The gas then flows down through the second pair of tubes C^1 , C^2 into the outlet bottom space C^3 , from which it is discharged through the valved outlet G^2 , the gas passing under the serrated edges G^4 of the depending casing G^3 , and then bubbling up through the water H^2 into the flue space H^3 , from whence it passes out through the discharge pipe H^1 . While the hot gas is thus passing through the vertical upcomer and downcomer tubes B^1 , B^2 and C^1 , C^2 , air, or air and steam, for the support of combustion in the producer E is also flowing through the circular casing A, in contact with the outer surface of the tubes, so that it becomes super-heated before entering the blower-casing or grate, while the gas becomes partly cooled. The baffle plate or air deflecting piece A^3 causes the entering air to take a regular upward and downward course amongst the tubes C^1 , C^2 , and B^1 , B^2 , before passing to the outlet J^2 , so that it comes thoroughly in contact with the tube surfaces, and thereby gets effectively heated before passing to the blower casing or grate.

An installation of the A. B. Duff by-product recovery plant, by the Gas Power and By-Products Co., of 38, Bath Street, Glasgow, for gasifying about 500 tons of fuel per day is shown in Fig. 86. Fig. 85 is a diagram shewing the arrangement of the Duff plant. The fuel is brought to the plant and emptied into the hopper A. It is delivered to the hopper C (which may hold sufficient for twelve hours' continuous working), by the conveyor B. The fuel is fed into the producer D, where it is gasified. The average rate of gasification in these producers is about 16 cwt. per hour, but the makers state that when required they will work up to 25 cwt., and still give good gas, but this slightly lowers the amount of by-products recovered.

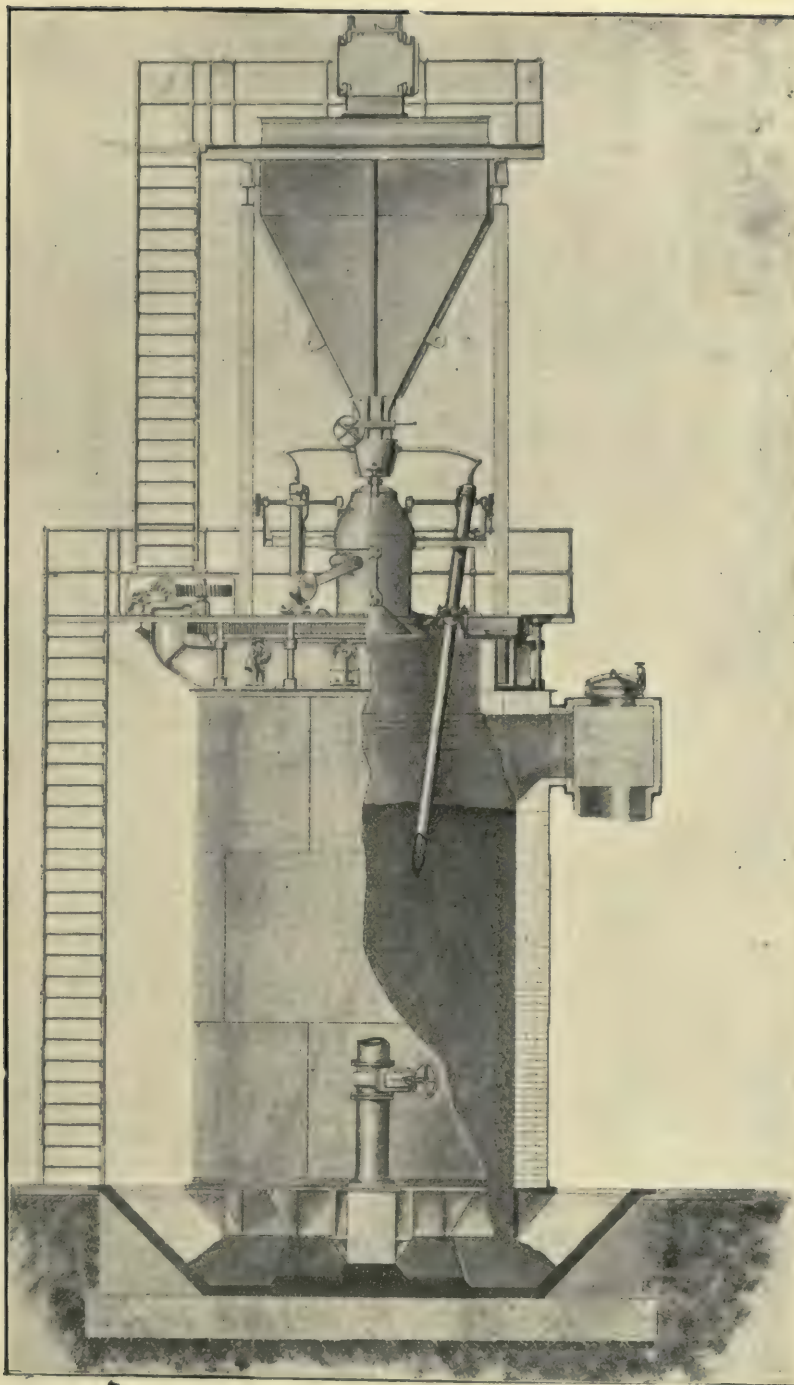


Fig. 87.—"A B DUFF, 1909," ROTARY TOP PRODUCER, BY THE GAS POWER AND BY-PRODUCTS CO.

The gas enters the superheater E (Fig. 85), which is so arranged that the hot gas is utilised in heating the air and steam blast on its way to the producer grate, where it is delivered under pressure. The gas, leaving the superheater, travels along the gas main to the washing apparatus F. This is a large horizontal chamber, water-luted at the bottom, in which the gas is subjected to sprays of cold water. By this method most of the dust and tarry matters carried in the gas is deposited in the chamber, which is so designed as to permit removal of any deposit with the minimum of trouble.

From the washing apparatus the gas passes to an acid washer, here shown as the tower G, in which it is treated with diluted sulphuric acid (passing continuously through the washer or tower) which combines with and fixes the ammonia in the gas. The liquor obtained from this washer or tower is run into the tank K, and is ready for feeding into the evaporating pan L, where it is boiled down to solid sulphate of ammonia. This pan is of the vacuum type, and heated by either live or exhaust steam, and may be continuous working. The box M on the bottom of the pan receives the salt crystals in a moist condition, and is so arranged that on discharge of the box the crystals are delivered into the centrifugal drying machine N, which thoroughly dries the salt, and it is then ready for the market. The gas is passed from the washer or tower G to the tower H, where it meets a descending stream of cold water, which further washes the gas, and cools it to any desired temperature.

Fig. 87 shows the "A. B. Duff 1909" rotary top producer, with mechanical poking, as made by the Gas Power and By-Products Co. This producer is specially adapted for gasifying and recovering ammonia from coking and inferior fuels. It is claimed that this design overcomes the difficulties arising from the swelling and coking together of such fuel in the producer, forming a mass through which the blast could not pass freely. The coke in this condition could not be properly gasified, and, in addition, air channels were formed, chiefly up the sides of the producer, so that undecomposed air passed through these channels, causing burning of the gas and ammonia in the top of the producer. The makers claim to have entirely overcome these difficulties, and that they can now obtain with coking coal all the economies due to the recovery of by-products which have hitherto only been realised when free burning coal has been used.

The Lymn By-Product Recovery Gas Producer.—Fig. 88 shows an 8,000 h.p. gas producer with ammonia recovery on the Lymn system, embodying the patents of Mr. Arthur H. Lymn, and made by the Lymn Chemical Engineering Co., Ltd., of Westminster Palace Cham-

bers, London, S.W. Fig. 88 shows the general arrangement of a 200 ton producer with ammonia recovery.

An ordinary blower or blowers are used to produce an air blast,

General Arrangement of a 200 Ton Producer Gas and Ammonia Recovery Plant. Rymin System.

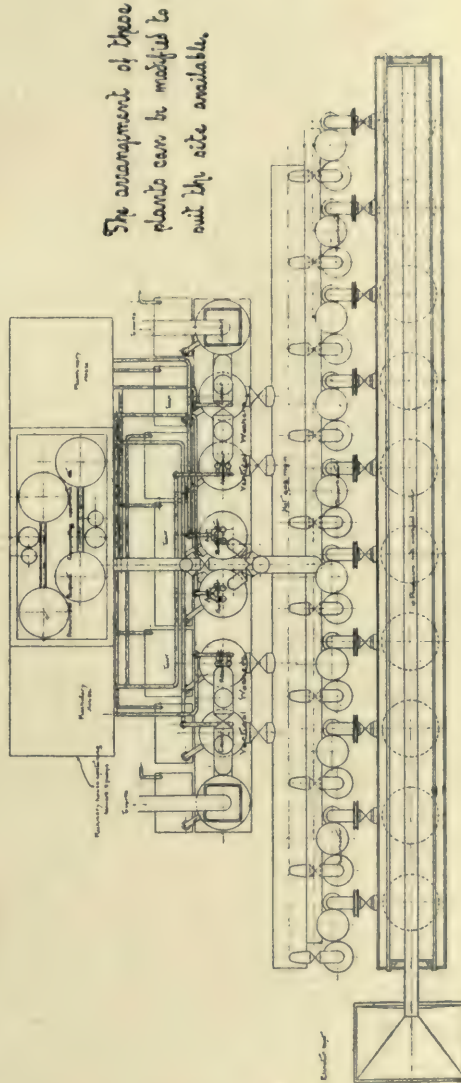


FIG. 88.

which passes through a vertical mechanical intensive air-saturating apparatus, in which it is brought into intimate contact with hot water (made hot by cooling the gas at a later point of the process). It thus

becomes saturated with steam, and after receiving a still further supply of steam it passes into the super-heaters, in which the air and steam mixture is superheated by means of hot gases leaving the producer. The air and steam mixture then passes to the grate of the producer, being superheated still further on the way by passing round the producer itself.

The producers, which may have a mechanical or non-mechanical grate, according to circumstances and the coal to be used, are provided with a water seal, from which the ashes may be removed, in the former case automatically and in the latter manually, without interfering in any way with the operation of the producers. In practice it is claimed that they need not be stopped more than once in one or more years, and then only for a couple of days.

The temperature in the by-product producers is comparatively low (due to the excess steam used), which has the result that the formation of clinkers is prevented. The ammonia formed, however, would be dissociated, were it not protected by the excess steam which is introduced for this purpose with the air blast in greater quantity than is decomposed. The hot gases containing the ammonia leave the producer at the top and pass into the superheaters, and superheat the air and steam blast which is passing in the opposite direction to the producers, and at the same time the gases are cooled, recovering a great amount of heat. On leaving the superheaters the gases, after passing through special dust separating apparatus, enter the vertical intensive mechanical ammonia absorber, where they are brought into intimate contact with an atomised solution of sulphate of ammonia, containing a small excess of sulphuric acid, which absorbs the ammonia thus produced. The solution becomes very concentrated, and requires very little evaporation for the crystallising out of the solid salt.

From the ammonia absorption apparatus the gases, after being freed from the ammonia, pass into the vertical mechanical intensive gas cooler, in which they are brought into intimate contact with cool water. The gas cooler and the air saturator work in one cycle, the water first passing through the gas cooler in order to cool the gas (the water being heated) and then into the air saturator in order to saturate the air, and again cool the water ready for the gas cooler. By this means a very large amount of steam is recovered from the gas.

From the gas cooler the gases pass to a second gas cooler, where they are brought again into contact with cold water. The gases are now sufficiently cool and clean for firing boilers or furnaces. If for use in gas engines the gases have to pass through one or perhaps two centrifugal cleaners, where they are almost entirely freed from tar, and from

here (after traversing a special water spray separator), the gases are passed through a dry scrubber filled with sawdust or wood shavings, in which the last traces of tar are removed.

The Lymn-Rambush Gas-Producer.—Figs. 90, 91, and 92 show the Lymn-Rambush producer, made by the Lymn Chemical Engineering Company, Limited. Fig. 90 is a perspective drawing, Fig. 91 is a sectional plan of the revolving grate with the ash-removing mechanism, and Fig. 92 is a vertical cross-section.

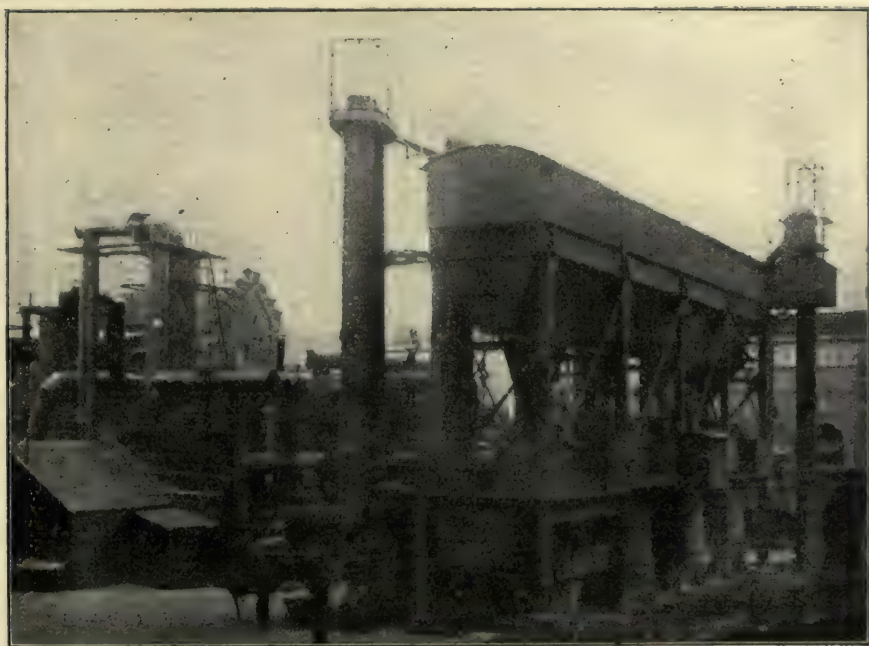


FIG. 89.—8,000 H.P. LYMN BY-PRODUCT RECOVERY GAS PRODUCER INSTALLATION.

For attaining the most profitable and constant gas results when using gas producers it is essential that the fuel bed be maintained in a good gasifying condition over its whole sectional area. In a properly operated producer the fuel rests upon a complete layer of ashes at the bottom having a constant depth and grade over the whole area. It is important that this layer be never allowed to clog or set solidly together, nor should temperatures become high enough for the ashes to fuse, and cause clinker, a failing which it is very difficult to avoid with many fuels in an ordinary hot gas producer.

In the Lymn-Rambush producer accordingly the whole of the ash zone is kept in a state of very slow but constant motion, which indeed is not confined to this zone, but extends also into the lower layers of the fuel zone above. This motion takes place in a horizontal as well as a vertical direction, whilst at the same time there is a slow but regular removal of ashes from all parts of the base of the producer proper.

In producers of the mechanical type previously built there has, in general, been only a single shovel, which removes the ashes at one part only, and this, it is held, causes an excessive local displacement of material from that side of the producer on which the shovel in question is fixed. The fact that the ash-bowl revolves does not eliminate this difficulty, because of its very slow speed of, say, one revolution in

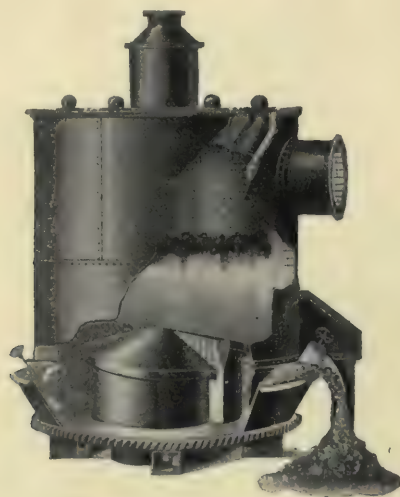


FIG. 90.—LYMN-RAMBUSH GAS PRODUCER: PERSPECTIVE VIEW.

one or two hours. The arrangement tends therefore to make the fuel bed more open immediately behind the ash shovel than over the rest of the sectional area, which involves a serious risk of uneven gasification. The makers claim, moreover, that a further serious drawback is to be found in many existing producers, *viz.*, that the apertures through which the air blast enters the lower zone of the producer are liable to become obstructed by ashes, owing to the pressure of the ashes against them. This results in considerable resistance to the passage of the air blast, and consequently to a reduction in the rate of gasification.

As will be evident from the figures, the producer is of the water-lute type, and the usual method of revolving the basin or bowl containing the ashes and water has been adopted. The ash-bowl, which car-

ries the grate, is suitably supported, and consists of an annular cast-iron turn-table, to the upper side of which is bolted the cast-iron side wall of the bowl, which has the shape of an inverted truncated cone. On its outer edge the turn-table is provided with a toothed ring, which works in conjunction with a steel worm suitably driven by means of a ratchet

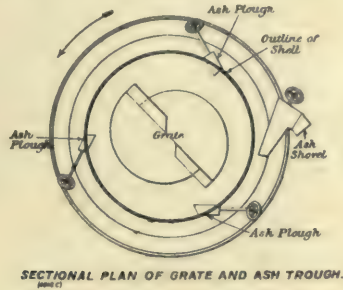


FIG. 91.

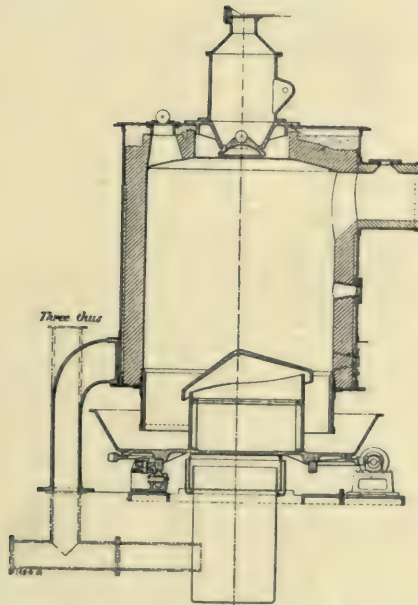


FIG. 92.

wheel and an eccentric, thus bringing about a slow revolution of the ash-bowl and grate.

As indicated in Fig. 92, or perhaps even better in the perspective view, Fig. 90, the producer grate or air distributor has the shape of two vertical half cylinders laterally displaced along their dividing surfaces. The top of each half cylinder has much the shape of a screw pro-

pellor blade, so that during each half revolution a vertical propeller-like movement is imparted to the ash and fuel in addition to the balanced eccentric movement caused by the two laterally displaced half cylinders. This combination provides the desired gradual but regular horizontal and vertical movement of the charge in the most simple manner.

The slots in the grate, through which the air blast passes into the gas producer proper, are placed in such a position that they can never become obstructed with ashes, as the air passes direct into the two wedge-shaped openings formed by the displacement of the half-cylinders. These openings in revolving move away from the ashes, which consequently remain extremely loose.

In order to secure uniform removal of the ashes from the whole circumference of the producer proper, simple ash-ploughs or scrapers are fixed upon the bottom of the producer, as shown in Figs. 90 and 91. These have the effect of loosening or ploughing up that part of the ashes immediately under the producer proper, and of displacing them into the revolving ash-bowl, which then carries them round until they reach the ash-shovel which discharges them into the ash-truck direct or on to the ground. It will be seen that although only one ash-discharge shovel is provided, the series of ash-ploughs remove the ashes continually and regularly from the bottom of the charge inside the producer, thereby maintaining the fuel bed always in a constant condition for good gasification.

Although at first sight it might seem as if the double-stage ash removal were a complication, it must be borne in mind that neither stage involves the use of any mechanical gear, and that the ash ploughs themselves, being attached to the base of the producer, operate quite automatically, and without the slightest manual attention. Moreover, their depth can be so regulated as to deal comfortably with any quantities and qualities of ash, without any of the difficulties attributable to a single stage ash discharge. The final result of this arrangement is that all grades of coal, even the waste dump heap of collieries, containing up to 40 or 50% ash, can be satisfactorily used, producing a good gas. The actual result of several months' night and day operation with a colliery waste of a coking nature containing 30 to 40% ash was a production per ton of fuel gasified of 110,000 cub. ft. of good gas, and when worked under ammonia recovery conditions 60 to 70 lbs. of sulphate of ammonia. It is stated by the makers of this producer that it is equally successful both with and without ammonia recovery, yielding maximum amounts of producer gas and ammonium sulphate respectively.

The Thwaite Gas Producer.—Fig. 93 shows the gas producer of B. H. Thwaite, as described in his British Patent No. 16,986 of 1902. A is the gas generating vessel or apparatus supplied with fuel by the elevator B. C is a directly associated physical gas scrubber or rough filter, D is a tubular recuperator through which the air to support the process of gasification is compelled under pressure to flow; in its passage through the nest of tubes it abstracts part of the sensible heat from the combustible gases generated in the vessel A. The heated air flows through the air mains E directly to the tuyeres F. The air is supplied in equal proportions to the tuyeres by the main G.

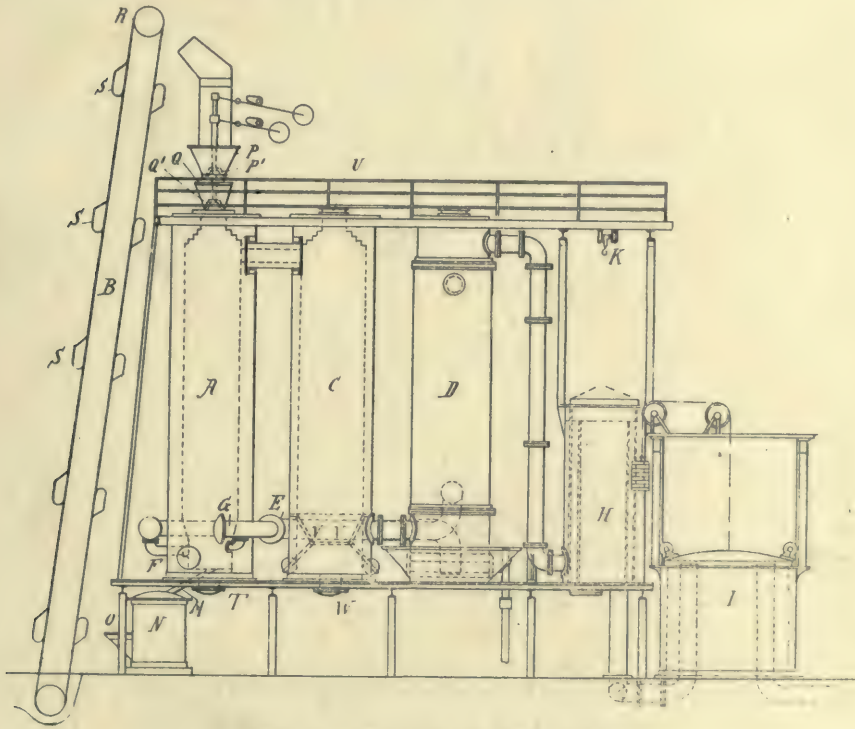


FIG. 93.—THWAITE GAS PRODUCER.

The recuperator vessel D is connected to the final physical purifier or filter H; in its forced passage through this purifier the combustible gas is thoroughly purified from suspended matter. This gas, purified and cooled, now enters the holder I. The movement—rising and falling—of this holder is made to regulate the flow of the air into the gas generating apparatus A, a simple by-pass valve opened or shut by the holder movement, if opened, allowing the air to flow back to the inlet of the fan or it may check the flow of the air.

The first three elements of the plant are preferably mounted on a raised platform; this arrangement facilitates the removal of the effluents, fused clinker or melted slag, from the generator vessel, and the dirty coke or other exhausted agent from the scrubbing vessel C. The arrangement of the tubular recuperator vessel also acting on the gas cooler, includes ready means of access to the interior to permit the removal of any deposit. The final physical purification element H consists in suspending an annular cage, charged or filled with a closely packed filtering agent that although porous requires that the gas to be purified shall only be able to flow through the resisting medium by the aid of a considerable pressure applied to the gas. Over the second purifying element H is placed an overhead travelling crane K that permits the purifying agent to be rapidly changed. When the plant is required to run continually the third or H element of the plant is duplicated, or as an alternative a holder of sufficient gas holding capacity to permit the change to be effected without involving the stoppage of the gas supply to the engine is provided.

Referring now more specifically to each element of the plant, it will be seen that the gas generator vessel A is very similar in design and construction, but on a miniature scale, to a modern type of iron-making blast furnace, and in some respects resembles a modern type of foundry or Bessemer iron melting cupola. Equi-distant from each other are provided water-cooled tuyeres, and in some applications the hearth, or the wall surrounding the zone of highest temperature, is water-jacketed. The inside surface of the hearth walls L is sloped at such an angle as will secure the uniform descent of the melted slag and fuel. An outlet orifice M is provided at the base of the gas generator A to permit the melted slag to run out of the generator vessel into another and externally placed receptacle N. This latter is arranged in such a way that the outlet channel can be readily cleared from obstruction, and the connection between the slag receptacle and the base of the generator vessel may be arranged in such a way that a liquid seal O is constantly established; the slag receptacle is covered as shown.

In order that the operation of charging the fuel and its associated lime stone shall be uniform, and that the materials shall be uniform, it is passed through a breaker to reduce both limestone and coke to nearly the same physical dimensions. They are then mixed along with the sand or clayey ore, and delivered to the skips or buckets of the elevator B. To prevent any serious proportion of gas from escaping during the charging operations, an automatic feeding device is employed, consisting of two charging bells, one superposed over the other. The fuel and limestone sand, etc., are charged into the upper bell chamber P, and

from this latter it falls into the lower chamber Q, the bell Q¹ of this latter being closed when the upper bell P¹ is open or dropped and *vice versa*, so that the gas under pressure is thus trapped, and therefore practically no loss of gas occurs during the fuel feeding operation. The sprocket wheel R of the elevator gearing actuates the charging bells so that they are lowered or raised in harmony with the movement of the elevator skips S. For instance, when the elevator skip S is depositing its contents into the hopper of the upper bell chamber P, the upper bell P¹ is closed, when the contents are discharged from the elevator skip or bucket and received by the upper bell chamber P, then its bell P¹ is lowered or dropped, by preference automatically, the material dropping into the chamber Q, the bell of which Q¹ is then also dropped, allowing the material to fall into the generator. In addition to the orifice M provided to enable the melted slag to escape from the generator, at the base of generator, and for the purpose of permitting ready access to the interior, may be provided a retort lid door T.

The second physical purifying vessel is equipped with a retort lid type of charging door U to permit the scrubbing agent to be readily charged. At the base of this vessel is provided a balanced grating V that supports the scrubbing agent; by merely tilting this grating the exhausted or dirty coke or other agent employed can be readily discharged from the vessel, falling as it does into the base of the vessel, from which it can be readily withdrawn through the door W, or a fresh supply of coke can be fed into the vessel through the retort lid U located at the top. The recuperator vessel D serves the double purpose of reducing the sensible heat of the combustible gas to the temperature of the atmosphere, and raising the temperature of the air employed in the gasification process so that the thermal efficiency of the process is claimed to be higher than that attained in ordinary generator gasification processes. In order to permit ready entrance to the base of the recuperator for cleaning the tubes, and to permit the deposits to be readily collected, and to provide a receptacle for receiving any moisture condensed from the gas the hydraulic bath is provided.

The Dellwik Water-Gas Producer.—Figs. 94 to 97 show the water-gas producer of Carl Dellwik, as described in British Patent No. 29,863 of 1896. In his specification Dellwik says: "By 'water-gas' is generally understood the gases produced by the decomposition of steam by coals, coke, or other suitable fuel at a white heat, and which may be considered as consisting mainly of a mixture of hydrogen and oxide of carbon. In this decomposition a large amount of heat taken from the coal is absorbed. If therefore the coals are heated, as is generally the

case, by blowing air into furnaces or so-called generators and not into specially heated retorts, then in order to again raise the temperature of the combustible material (coke, coals or other fuel) which has fallen in consequence of the decomposition of the steam, that is to say during the period of the production of gas, it becomes necessary to shut off the admission of steam and to introduce air instead, in order to revive the fire. In this manner in the production of water-gas by generators, the periods of production of gas (by the introduction of steam) and of the introduction of air to revive the fire, constantly alternate. In the hitherto known processes for manufacturing water-gas the object in view has been to supply to the coals arranged in layers of great height, during the air supply period such a quantity of air as to cause the coals to burn incompletely and produce oxide of carbon, but not to effect a complete combustion, resulting in the production of carbonic acid; the effect aimed at being to produce during the air supply period a combustion product containing (together with free nitrogen) as much carbonic oxide as possible, combustible and suitable for heating purposes, or in other words, to produce so-called producer-gas. The hitherto employed processes for the manufacture of water-gas have therefore been characterised by the combustion products obtained during the air supply period (the so-called producer-gas) containing more carbonic oxide than carbonic acid. This fact constitutes a distinct demarcation line separating the processes hitherto used from the new method which forms the subject of the present Patent, and the aim of which, on the contrary, is to obtain during the air supply period combustion products containing a larger quantity of carbonic oxide. The new method does not therefore aim at the production, during the air supply period, of a combustible producer-gas, that is to say a gas containing chiefly carbonic oxide; its aim, on the contrary is to considerably increase the production of water-gas, the heating value of which is four times greater than that of the best producer-gas. The practical advantages of this new process are very great. While hitherto in order to produce water-gas during a period of four or at the utmost six minutes, an air supply period of fifteen to twenty minutes was required, to raise the temperature of the fuel to the extent necessary for the decomposition of steam; experiments with the new process have shown an air supply period of three to four minutes to be sufficient to render possible a gas production period of twelve to seventeen minutes, before a fresh introduction of air becomes necessary. It is well known that carbon, when burning so as to form oxide of carbon, develops scarcely one-third of the heat capable of being developed if the same quantity of carbon is burned so as to produce carbonic acid.

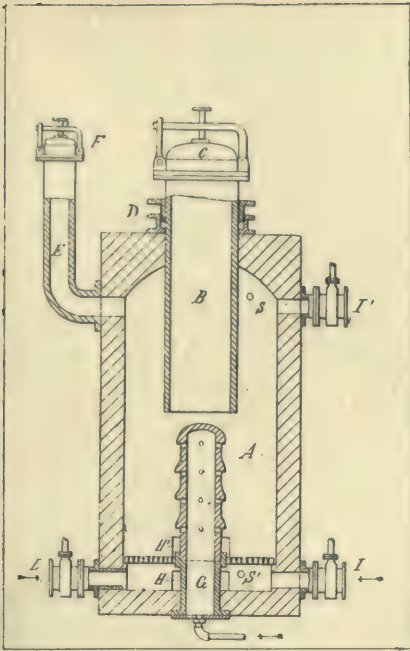


FIG. 94.

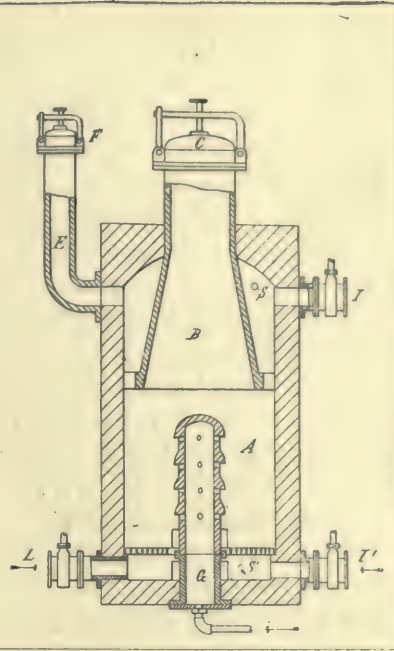


FIG. 95.

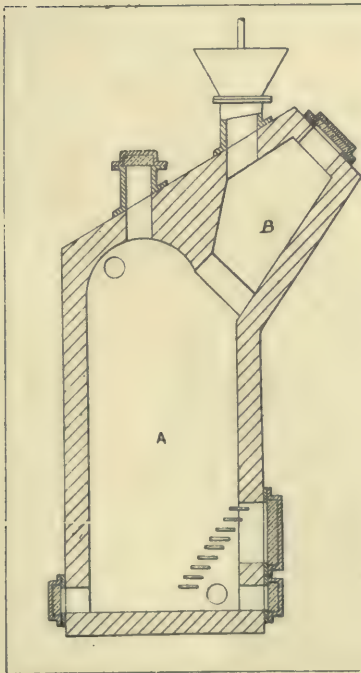


FIG. 96.

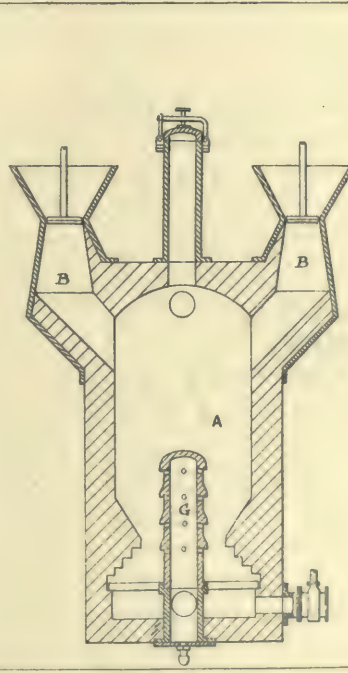


FIG. 97.

"The object of the present invention is to conduct the process so as to produce during the air supply period a gas containing as much carbonic acid as possible and as little oxide of carbon as possible, an aim directly opposite to the object of all the various processes hitherto employed.

"By the new method, therefore, a much greater amount of heat is generated and an adequate saving of fuel effected, the volume of water-gas produced with the same amount of fuel being doubled in consequence while the time of the air supply period is correspondingly reduced and that of the gas production considerably increased.

"The purpose of the present invention is attained by regulating the proportion between the height, volume and size of coal, coke or other suitable fuel employed and the amount of air admitted in such a manner as to cause a complete combustion of carbon to carbonic acid. By properly proportioning the air supply to the height of combustible fuel combustion products are with certainty obtained during the air supply period, which to the absolute or nearly absolute exclusion of carbonic oxide, contain only or chiefly carbonic acid, besides nitrogen. The exact proportion varies of course in accordance with the nature of the combustible material employed and other circumstances and must be determined experimentally in each separate case. For coke of average size, for instance, it has been found that with an air pressure of 210 millimetres water under the grate and with 60 millimetres above the layer of coke (that is to say with an absolute pressure of 150 millimetres in the layer of coke), the height of the latter should be about 1200 millimetres in order to obtain combustion gases nearly free from oxide of carbon and containing about 20% of carbonic acid. Such a height of coke is also sufficient to completely decompose the steam admitted during the period of the production of gas. According to whether it is desired to use the fuel in layers of greater or less height, the air supply is increased (by means of an increase of pressure) or else diminished so as to produce in each instance during the air supply period a complete combustion of the fuel to carbonic acid. For this end it is equally practicable to regulate the height of coke or other fuel in accordance with a given pressure of the air supplied, as to make the reverse adjustment, *i.e.*, to proportion the pressure of air in accordance with a given height of coke or other fuel; but in all cases experiments will indicate a certain definite proportion according to which for a certain pressure of air supplied a certain definite height of coke or other fuel must be maintained. For the application of the process according to the present invention any water gas apparatus may be used, if only the height of coke is regulated by frequent and regular charging in of fresh fuel so as to keep it at a certain and approximately

constant height which shall ensure complete combustion to carbonic acid according to the pressure of the air supplied. It is however preferable to use an apparatus in which the height of coke is maintained constant in an automatic manner. For the determination of the most suitable height corresponding to a given pressure of the air supply, it is advisable to use a device having an adjustable coal receptacle." Such apparatus is represented in Fig 94. A is the generator provided with a coal receptacle B; this coal receptacle passes through a stuffing box D placed on the cover or top of the generator. Under the grating is arranged the main air inlet L and the gas outlet I and steam inlet S¹. The generator is also provided with cleaning doors H and H¹. In the upper part there is an outlet pipe E for the combustion products, which pipe is closed during the period of the production of water gas by the valve or cover F. I¹ is an outlet for water gas and S is a second steam inlet. The coal receptacle B is charged with coal, coke, or other suitable fuel and then closed by the door or cover C. The air inlet is arranged under the grate and preferably continued into the layers of fuel by an appropriate pipe G provided with numerous air nozzles or apertures, so that a through contact of the air with the whole mass of fuel is obtained. When the proper height of a given description of fuel for a given amount of air pressure has been determined by testing by means of the adjustable receptacle B shown in Fig. 94, the cast iron receptacle is advantageously replaced by one made of fire bricks, as illustrated in Fig. 95.

CHAPTER IV.

SULPHURIC ACID PLANT.

It has been made clear already in preceding sections that it is not the intention of the author to enter into any detailed discussion of the theory of design or construction of any class of chemical plant dealt with in this manual. This limitation applies very materially in the case of sulphuric acid plant, and indeed it would be presumptuous, in such a volume as this, to attempt to deal with the subject on any other lines than those laid down in the preface. The whole subject is very fully covered by Lunge in his "Sulphuric Acid and Alkali," and the general problems of vitriol plant design and construction are also treated at some length, with detailed working drawings of actual installations, in "Chemical Works: their Design, Erection, and Equipment" (Dyson and Clarkson, London: Scott, Greenwood and Son), to which the reader is referred. In the last mentioned volume, the general considerations involved in the choice of a site for the plant, the laying out of the works, the general design of a typical vitriol plant, the construction and erection of the chambers, with their details, the position of the burners, the construction and erection of the Glover and Gay-Lussac towers with houses, etc., are all dealt with by Mr. S. S. Clarkson, Assoc. M.I.M.E., in the light of a long experience of vitriol plant design, and his set of actual working drawings covers all the points to be considered in the erection of the usual chamber plant. The present chapter will consist, therefore, of a review of the recent trend of development, with a summary of such of these developments as have come particularly to the notice of the author.

The question of the modern development of the chamber process is well summed up in the forty-ninth Annual Report of the Chief Inspector of Alkali Works, in which the Chief Inspector says:—"For a process

dating from the end of the 18th century, the chamber process for manufacturing sulphuric acid from sulphurous acid, vapour of water, and air, with the aid of oxides of nitrogen, is noteworthy as still affording a field for variety of procedure and modification of plant for which substantial advantages are claimed. The form of burner to be used for producing the sulphurous acid required, the use of fans for regulation of draught, the use of water sprays instead of steam, the design and arrangement of towers, the design and arrangement of chambers, the methods of supplying and preventing loss of oxides of nitrogen, and the proportion of "tower" space to "chamber" space, have all received attention in recent years, and fresh proposals are frequently brought forward by inventive minds for the judgment of the manufacturers. In many cases these newer methods have been and are being adopted, and their effect is being watched with interest."

Bulletin No. 283 of the U.S. Department of Agriculture, by W. A. Waggaman, contains an excellent review of recent chamber developments from which the following summary is quoted.

The efficiency of a lead-chamber plant is measured, first by the amount of chamber space required for each pound of sulphur burned in 24 hours and the amount of acid (50° or 60° B.) made therefrom, and, second, by the amount of nitre consumed or lost in the production of this acid.

Practically all sulphuric acid authorities agree that, provided the gases are present in the proper proportions, the two most important conditions necessary for efficient production are a thorough mixing of the gases and the control of their temperature.

The importance of the first of these conditions is self-evident, since in order to bring about complete chemical reaction the reacting substances must be in intimate contact with one another. The second condition is important, because too low a temperature lessens the chemical activity of the gases, while a temperature above 100° C. prevents the condensation of water, which it is claimed is necessary to bring about the decomposition of nitrosulphuric acid, an intermediate compound formed from the oxides of nitrogen in the system.

Numerous schemes to control these conditions have been devised, some of which have features of considerable interest and practical importance, and several that have been tried, apparently with some success, are described below.

Walter and Boeing advocate the use of several hollow acid-proof partitions built across the chambers, and so arranged that the gases enter the compartments through large holes near the bottom, and are

discharged from holes near the top. Numerous other holes allow the admission and exit of the gases, thereby causing them to mix intimately without seriously interfering with the draft. Because of the doubtful stability of these inner walls and the serious damage caused on their collapse this method is no longer used. Gossage, as well as several other investigators, proposed filling the chambers with coke, so that the gases would be obliged to work their way through the interstices, and thereby become thoroughly mixed. This scheme, however, has been abandoned because of the impurities introduced into the acid by the coke, and the tendency of the coke columns to press against the lead walls, causing them to bulge and even break. The lack of any cooling device in this process also caused excessively high temperature in the chambers.

Verstrat's plan is similar to the above, except that stacks of bottomless stoneware jars filled with coke are used. The oxides of nitrogen are supplied to the system by allowing nitric acid to trickle down one of the stacks.

In Pratt's process, which is much used in the Southern States, the gases are drawn through the first chamber by means of a fan, then through a tower packed with quartz down which flows dilute sulphuric acid, and finally they are re-injected into the front of the first chamber by means of the same fan. This circulatory system seems quite efficient, and a number of plants where the process is employed are operating on less than 9 cubic feet of chamber space per pound of sulphur burned in 24 hours.

Meyer's tangential chambers are designed both to mix and to cool the reacting gases at the same time. The chambers are cylindrical in form, the first having water-cooled lead pipes suspended around the circumference. The gases are admitted at a tangent near the upper part of the chamber walls, and are discharged from outlets in the centres of the chamber floors. The gases are thus given a spiral motion which tends to mix them thoroughly, while the water-cooled lead pipes reduce their temperature. There are three installations of this type of plant in the United States. One at least is reported to have given great satisfaction.

Hartmann obtained an increased yield of acid in the lead-chamber process by placing vertical, air-cooled lead pipes in the chambers. The chamber bottom is turned up around the lower ends of these pipes, forming hydraulic seals, and thus obviating the necessity of joints in the bottom of the chamber.

Blau proposed to cool the gases in the first chamber by injecting a spray of cool sulphuric acid, and in order to obtain the optimum yield

of acid from the gases in the subsequent chamber their temperature is raised by injecting sprays of warm sulphuric acid.

Falding's process has for its object the segregating of the active gases in a system. To accomplish this he employs a chamber the height of which is approximately one and one-half times greater than its horizontal dimensions. The burned gases, after passing through the Glover tower in the usual way, are introduced either near the top or lower down on the chamber side. Since the fresh gases are hot, not only because they have recently issued from the pyrites burners, but because of the reactions taking place between some of the constituents, they collect in the upper part of the chamber in a relatively active layer. As the reactions subside the spent gases gradually cool, and settle to the bottom of the chamber, where they are withdrawn. Falding claims that by using the high chamber a zone of great chemical activity is always maintained in the upper part of the chamber, and that the spent or inactive gases, which in ordinary chamber systems act as diluents, are continually being removed from the active zone. It is also claimed that much less chamber space is required to complete the reactions by this process, so that even where large volumes of gases are handled each chamber is a unit in itself, being connected directly with the Glover tower instead of in series as in ordinary chamber systems. A number of plants in the States are equipped with chambers of this type, and it is reported that the process is commercially successful. The main objections to the Falding system, in the opinion of the writer, are first, that no provision is made for obtaining an intimate mixture of the gases other than the preliminary mixing brought about in the Glover tower, and, second, that no adequate means is provided for the condensation of the acid mist formed by the reaction.

The most widely used method of mixing and cooling the reacting gases is by means of intermediate towers containing plates, tubes, or baffles of some acid-resisting material cooled either by water, air, or dilute sulphuric acid. A number of different types of towers have been designed, but mention is here made of only a few of the better-known designs.

Lunge's plate tower consists of a shell of lead either cylindrical or angular in form, and filled with a series of perforated plates laid horizontally. Each layer of plates is supported at some distance above the other by bearers in such a way that every plate is independent of the others. The plates are so constructed and placed that the holes in those of one layer do not come directly above the holes in the next layer below. Dilute sulphuric acid is allowed to trickle down the tower, splashing from one layer of plates to another, and meeting the hot chamber gases as they

wind upward through the tower. The film of dilute acid over the plates presents an immense cooling surface to the gases, and at the same time furnishes the water necessary for the decomposition of the nitrosulphuric acid. The formation of this latter compound is, according to Lunge, a necessary link in the chamber process.

Gilchrist's pipe-column system consists of lead towers (3 or 4 feet across and 15 feet high) having corrugated lead tubes, open at both ends, running through them horizontally like a steam boiler. The sides of the towers are boxed in with boards so as to form an air shaft which terminates in a flue at the top. The chamber gases, together with water vapour, enter the pipe columns at the sides near the bottom, and work their way upward through the towers. Contact with the air-cooled corrugated tubes condenses the sulphuric acid, which then drips in showers from one series of pipes to another, and frees the oxides of nitrogen, restoring them to the system. The gases issue from the top of the towers and enter the next chamber, from which they are drawn into another series of pipe columns, and so on through the system till their oxidation is practically complete.

While some of the methods just described are designed to cut down the amount of chamber space required, none of them, with the exception of Falding's process, reduces the initial cost of erecting an acid plant; for, while less lead may be employed in constructing the chambers, the expense of the cooling and mixing towers more than offsets the saving in chamber materials. Another objection to most of the accelerating devices discussed above is that in order to mix the gases thoroughly they must be drawn or forced through small openings, or made to pursue a meandering course by means of baffles or some acid-proof packing material in the towers. Under such conditions dust or impurities may clog the apparatus, choking off the draft, and making it necessary to clean out the tower or chamber before operations can be resumed. Moreover, the collapse or disarrangement of the packing material within the tower may cause even more serious trouble.

Dr. O. Nagel (*Zeitschrift für Angewandte Chemie*, 1912, p. 1220) observes that one of the first conditions for the successful execution on a commercial scale of reactions between gases is to secure perfect mixing from the beginning. In the chamber process for manufacturing sulphuric acid by the usual system there is clearly but little chance of a perfect mixture of the interacting gases in the chamber. The chambers are very large, and the gases enter them at different points, and with different speeds. It is no wonder, then, that plans have been suggested for remedying this, and thereby securing a far greater yield (the Gay Lussac and Glover towers are, in fact, merely devices for

lessening the waste due to imperfect mixing in the chamber). These plans may be classified as follows:—

(1) Alteration of the shape of the chambers, and devices for making the gases enter it with a spiral motion. Thus we have the tangential chambers of Meyer (D.R.P. 101,376). This device had some success, but in view of the height and size of the chamber, the degree of admixture still remained far from what it should be.

(2) The use of obstacles against which the entering gases strike, whereby mixing is promoted, and also the condensation of the chamber-fog. This idea is embodied in Lunge's plate tower (D.R.P. 35,126, 40,625, 50,336). This system has made it possible to replace all the chambers except the first by towers. Attempts to replace all the chambers by towers have so far failed. For a daily output of 7,200 kg. of H_2SO_4 with two towers, a chamber of 1,064 cubic metres is necessary.

(3) Mixing with a steam blast (as everybody knows, steam must be present in the chamber, and the reactions signify its absence in the usual manner by the formation of the dreaded chamber-crystals). Hence there is a danger if steam is used for stirring up the gaseous contents of the chamber that there will be too much of it, as Scheurer-Kestner (D.R.P. 15,252) found to his cost.

Richter also proposed to use injectors, but for the purpose of circulation only, sucking the gases continually from the lower part of the chamber, and returning them to it at the top. His apparatus, however, only circulated the gases near the walls of the chamber. As might have been expected, the gases in the middle of it were unaffected. Besides, as might also have been expected, the current of gas through the chamber was checked to such a point that the sulphur dioxide from the burners could hardly get into the chamber. The solution of the problem seems to be to secure a proper admixture of the steam, sulphur dioxide, air, and oxides of nitrogen, before they enter the chamber.

Dr. H. von Keler (*Zeitschrift für Angewandte Chemie*), in a review of progress in the heavy chemical industry in 1911, gives references to a number of German Patents dealing with modifications in the lead chamber process, having as their object the increase of the production with the smallest chamber capacity and nitre consumption. Of these may be mentioned Moritz's construction of the lead chamber (German Patent, 235,800), which facilitates radiation and effects economy in material. Rabe (German Patent, 237,561) passes the gases into the chamber in such a manner that the double spiral motion pointed out by Abraham may be induced or increased. The Opl tower system has been found satisfactory in the I. Oesterreichischen Sodafabrik at Hruschau, its principal

advantages being low initial cost, the small ground space required, and the easy control of manufacture. Wentzki (German Patent, 230,534) has improved his cylinder apparatus, the reaction chamber being no longer rotatory, but fixed, and provided with suitable stirring appliances. New filling materials have been proposed by Pavlitzek (German Patent, 229,999), and by Scherfenberg (German Patent, 239,072).

Reusch (*Chemiker Zeitung*) in the course of a lengthy review of the mineral acid industry, observes that in vitriol manufacture, mechanical burners have made great headway, especially in England and Austria. The horizontal type (French Patent 451,182) seems to give better results than the vertical type. It naturally requires less power. Rotary burners for getting the high temperature required for zinc blende are proposed by K. Eichhorn. The invention of R. Celarius (German Patent, 263,941), for using the waste heat of the burners for raising steam, deserves mention. The patentee suggests that all the steam required for working the machinery of a sulphuric acid factory may thus be obtained. Several inventions are concerned with the purification of the sulphur dioxide before it enters the chamber. The Societe Belge Koerting sprays water into the gas as it leaves the burner, thereby entangling dust and other impurities. The Graffich von Landsberg-Velen and Gemenische Fabrik passes the gas through an oxidation tower to free it from arsenic, then through a denitrating tower, and finally through water-scrubbers into the chamber. Klason and Mellquist have found that if the gases are passed through a close asbestos filter any selenium present is stopped, and can be afterwards obtained from the filter. There were in Germany, in 1913, eighty-six concerns working with lead chambers, and 17 with both lead and contact chambers. In Belgium there were 26 concerns with lead chambers having a total capacity of about 400,000 cubic metres. The chief improvements in the chamber process have consisted in the introduction of draught regulators, in using more water spraying, and in increasing the size of the Gay-Lussac towers. The methods of building and supporting the chambers have been greatly improved by Kalinowsky (German Patent, 260,991), Hartmann, Barth, and others. Alterations in the shape of chambers have also been proposed. For instance, Littman makes the second half of the first and second chambers lower than their first halves, to make up for the decreasing activity of the gases as they cool and move towards the Gay-Lussac tower. Partitions have been used for the same purpose. Parent's French Patent (449,035) provides for returning acid from the last chamber to the first chamber in order to get higher concentration in the chambers themselves. Petersen uses two Glover towers, one above the other, instead of using a single

tower of double the usual height. To accelerate the reactions in the chamber, Davis proposes to work splashers to throw about the weak acid, and keep it in circulation. Burkhardt proposes to replace chambers by towers filled with a double column of troughs, the gases entering at the bottom. Other forms of towers have been tried by Hoefling. The towers are often lined with acid-proof bricks, widest at the surface which comes lowest in the lining, so as to prevent the acid trickling down from getting at the mortar between the courses.

Hugo Petersen (*Zeitschrift für Angewandte Chemie*, 1912, pp. 762,765), in comparing the tower and chamber systems, quotes the following data:—E. Hartmann (*Zeitschrift für Angewandte Chemie*, 1911, p. 2302) states that for a production of 18 tons of 60 Bé. acid, 4000 cub. metres of air were required to lift the acid to the towers. About 1200 tons can be compressed 15 metres high, or 200 tons for each of the six towers used, every 24 hours. This is more than 10 times the daily production, whilst in the chamber system twice the daily yield is generally the figure. T. Meyer states for his tangential system a higher cost of plant and greater ground space than for the tower system; but for the Falding high chamber system both cost of plant and ground area are lower.

Hartmann gives the ground space required for the tower system for the daily production of 18 tons of 60 Bé. acid, as $40 \times 8 = 320$ square metres. In the Falding system this is:

| | |
|-------------------------|----------------------------------|
| 35 tons of 50° Bé acid, | $24 \times 19 = 456$ sq. metres. |
| 70 „ „ | $24 \times 36 = 864$ „ „ |
| 105 „ „ | $24 \times 53 = 1272$ „ „ |

Moreover, there are two conditions that favour the question of ground area, namely: (1) the Falding chamber forms a reservoir not present in the tower system; and (2) in addition to the place for the machines, coolers, etc., there remains free space under the Falding chambers for the storage of materials.

For Falding plants of different sizes, for a daily production of 7 kilos. per cub. metre, the cost of erection, including architects' fees, licences, plant for cooling water, boilers, condensing plant, hard-lead ventilators, pulsometers, and nitric acid appliances, but not including ground, kilns, plant for electric power and light, etc., would be:

| | |
|---|------------|
| For a plant for 35 tons of 50° Bé acid daily, | 183,000 M. |
| „ 75 „ | 326,100 „ |
| „ 105 „ | 422,900 „ |

Lead is put down at 40 M. per 100 kilos., and the thickness of the walls of the different parts are taken to be as follows:—

| | |
|--|-------------|
| Glover tower, | 8 to 10 mm. |
| Cooling and Gay-Lussac towers, | 4 mm. |
| Chamber and cover, | 4 mm. |
| Chamber walls, | 3 mm. |
| Pipe connections from the Glover to the chamber, | 5 mm. |
| Pipe connections from the chamber to the towers, | 4 mm. |
| Pipe connections between the towers, | 4 mm. |

If now these figures are compared with those for the tower system of equal capacity, and all the different considerations are taken into account, it cannot be said that the Falding system, even with an efficiency of 7 kilos. per cub. metre, costs more to erect than the tower system. Coming to the cost of working, these will be the same in both systems, so far as production of sulphur dioxide, consumption of nitre, labour and repairs, and general administration expenses go. The figure for the amortisation and interest will also be about the same, or at any rate will not be unfavourable for the Falding system. The cost of power, however, differs in the two processes, especially where the raising of the acid comes into consideration. In the tower system five times the amount of spraying acid has to be raised, and this will cost 11,000 M. in the case of a plant producing 35 tons per day of 50° Bé acid. Hartmann considers that an advantage in favour of the tower system is that the expenses of labour and management may be less, but it is pointed out, in disagreement with this statement, that the efficiency of the tower system depends to a very great extent upon exact and careful methods of working.

Dr. G. Schliebs (*Chemiker Zeitung*), discussing the question of the substitution of towers for lead chambers, points out that all the suggestions embodied in the many patents taken out during recent years may be divided into two classes. One of these is concerned with special methods of circulation, the other with particular methods of construction, whereby the interaction of the gases is quickened. In order that a tower may be adopted as a substitute for the lead chamber, it must not only have all the advantages of the latter, but other advantages not possessed by the lead chamber. The recognised advantages of the lead chamber are that it ensures complete mixture of the sulphur and nitrogen oxides, and air and steam provides for the escape of the heat produced by their interaction without requiring any special means, and produces differences of temperature inside the chamber. None of the proposed systems combine these three advantages. Dr. Schliebs thinks he has solved the double problem of doing this and adding the other advantages which, as he himself admits, must be secured if the new system is to compete with the lead chamber

and catalytic processes. He considers that the shape of the towers (in transverse section) and the nature of the packing, are both immaterial so long as the towers are higher than they are wide, and lined with lead or other suitable material, and comparatively small. As regards the mixing of the gases, if the gas circulation in a lead chamber of the usual size (from 1,000 to 3,000 cubic metres) is compared with that in the corresponding tower of one-tenth of the capacity, the following conclusion as regards the systems is reached.—In the lead chamber any given portion of gas stays a considerable time, say 30 to 60 minutes. During this time it is in active motion, so that the fresh gases arriving from the burners, etc., are kept mixed with the excess of those which have already taken part in the formation of oil of vitriol. Through the tower above specified the gases pass in a few minutes, and despite the mixing caused as they work their way through the packing, there is no time for the fresh gas to mix properly with the above-mentioned excess of the gases which have already interacted. As regards the cooling advantages hitherto offered by the tower substitutes, and which are secured in the lead chamber process by mere radiation, we find that a small tower filled with flint, coke, or any other packing offers a very small radiating surface, and the less in proportion the greater the transverse section. Finally, as regards the temperature differences, we have in the lead chamber a continual heating up in the inner parts of the chamber by the arrival of the fresh gas, and this is a very favourable factor, as every sulphuric acid maker knows. Now, with every tower hitherto suggested as a lead chamber substitute, no matter how it may be supplied from the sprinklers, the gases keep about the same temperature throughout. There may be a slight fall of temperature from the inlet to the gases, but there is no warming of them again as there is in the chamber. Dr. Schlieb's invention is to put behind the substitute tower an acid separator so arranged that when required it allows the gases to pass back again, and go for a second time through the tower. In this way the mixing of partly used and unused gases is made perfect, the cooling is easily effected by air or water cooling, and any temperature differences desired can be obtained, for the gases are cooled in the separator, and heated again when they enter the tower. So far the advantages of the lead chamber may be said to have been secured. As regards the essential extra advantages, these are:—The production of each substitute tower can be adjusted within wide limits. As the gases can be put through the tower again and again, the reaction between them is total, and sprinkling of the packing with acid at the top of the tower may be considerably cut down or omitted altogether, and Glover and Gay Lussac towers becomes super-

frous. The reaction being made complete, the consumption of saltpetre and sulphur (or pyrites) is cut down to the lowest possible point.

In dealing with particular details of vitriol plant, it should once more be observed that the arrangement of what follows in this chapter is that of convenience for sectional publication.

The Packing of Vitriol Towers: "Metalline" Bricks and Tiles.—One of the best known and most effective of tower packing materials is the "Metalline" ware of the Buckley Brick and Tile Co., Ltd. For nearly forty years now "Metalline" tower packing materials have been in constant use in a very large number of vitriol works, and with uniformly satisfactory results. In the *Chemical News* of February 2nd, 1882, Mr. John Morrison, of Newcastle, is reported to have spoken at the general meeting of the Newcastle-upon-Tyne Chemical Society as follows:—"Some two years ago I was entrusted with the erection of sundry plant for the Connah's Quay Alkali Company, which included a pair of Glover towers, but when the manager (Mr. Steedman) seemed inclined to try 'Metalline' bricks for their lining, I must confess I was at first very much opposed to what appeared to me to be a serious risk. After, however, carefully examining the Buckley Brick and Tile Company's process of manufacture, and after Mr. Steedman had boiled a Metalline brick for two months continuously in sulphuric acid, and found that no perceptible action had taken place, my prejudices gave way, and bricks and blocks of that material were finally adopted, not only for lining, but also for the arches of the towers. On November 29th, 1887, Mr. Steedman wrote:—"A few months ago I had an opportunity of carefully examining the Metalline tower lining supplied for Gay-Lussac towers at Connah's Quay Alkali Works in 1879, and I found that they showed very few signs of giving way or having been acted upon, and had they continued working would have lasted for many years to come. The test of the bricks only showed 3% acid."

Of the many tower-packing materials introduced by this well-known firm during recent years, one of the best is Mackenzie and Gibson's patent tiles, shown in Figs. 98, 99, 100, and 101, for Glover, Gay Lussac, hydrochloric, intermediate and other towers and structures for bringing gases and liquids into contact. Fig. 98 shows the upper and Fig. 99 the under side of these tiles. Fig. 100 is a section showing how the tiles work, and Fig. 101 shows the tiles packed. The great efficiency of the tiles is due to the fact that liquid runs over the upper side and clings to the under side till it reaches the serrations, where it is broken up to meet the ascending gases, as shown, and as the whole

surface of each tile is covered with a moving film of liquid, deposits are almost impossible, whereas in many other forms of packing there are either rectangular or flat surfaces or recesses, which induce sediments to lodge and choke up the passages. Fig. 102 shows the "Metalline" arch blocks which are used in many towers, as by the use of these blocks the table can be more easily built on them than on ordinary brick arches.

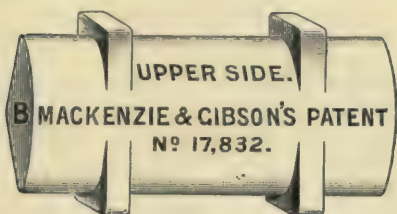


FIG. 98.

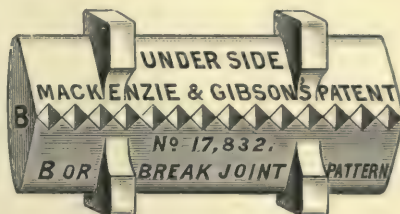
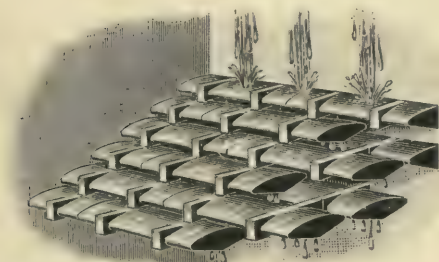


FIG. 99.



MACKENZIE & GIBSON'S PATENT, N° 17832

FIG. 100.

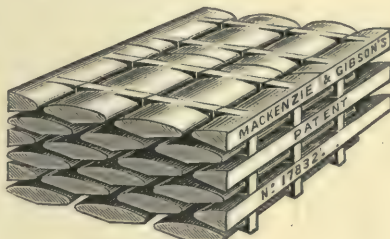


FIG. 101.

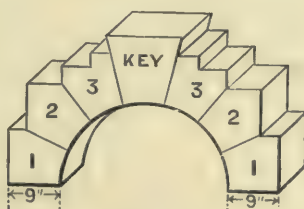


FIG. 102.

Fig. 103 shows the packing of a Glover tower with the tiles. The tower illustrated was erected in May, 1907, and was the first in which the tiles were used. Being experimental, it was not considered wise to use them from bottom to top, but the results attained clearly show that had more tiles been used throughout, results would have been even better. The tower dealt with all the gas from $10\frac{1}{2}$ tons of 50% pyrites in twenty-four hours, and has done so continually without the slightest

diminution of draft. The acid going down the tower in twenty-four hours = 40 tons nitrous acid containing 30 oz. of nitrate of soda per cube foot, and 16 tons of 120° Tw. chamber acid. The whole of the acid

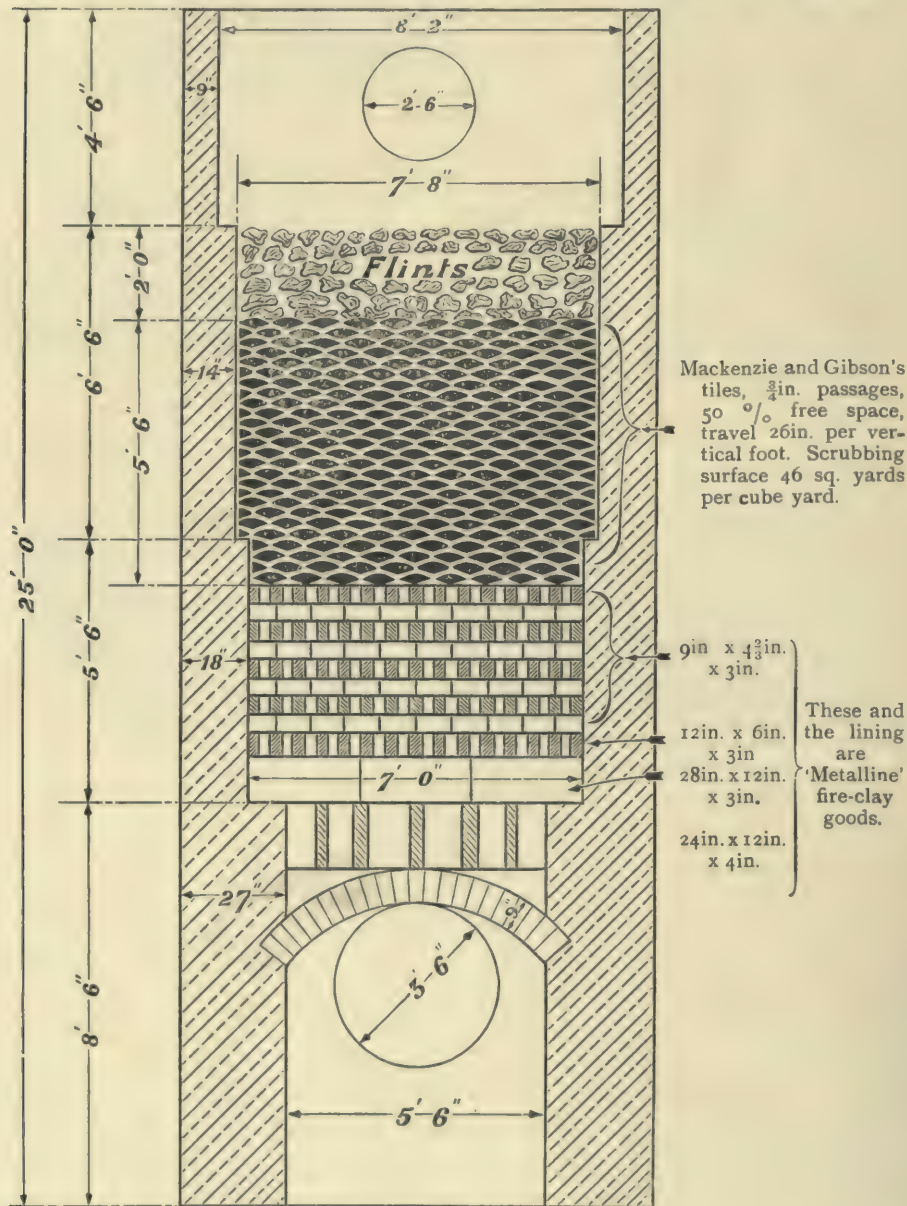


FIG. 103.

leaving the tower is 140/142° Tw., and is absolutely denitrated. The firm who erected this tower, and are still working it, have erected six similar ones.

Nielsen's "Propeller" Filling.—Nielsen's "Propeller" filling is illustrated in Figs. 104 to 108. In the Patent Specification relating to this material (British Patent No. 26,269 of 1913, Nitrogen Products and Carbide Co., Ltd., and Harald Nielsen) the inventors observe that one of the most important factors in the modern acid and other like industries is the so-called tower, whether this is used for absorption, reaction, mixing, or cooling purposes. In the acid industry such a tower generally consists of an outer casing or shell of suitable material, and a filling material,

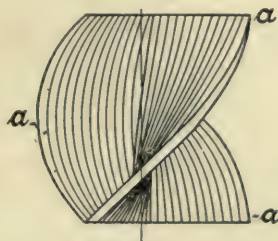


FIG. 104.

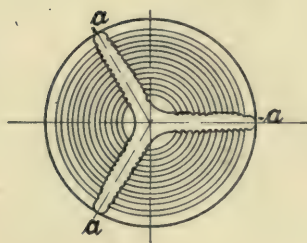


FIG. 105.

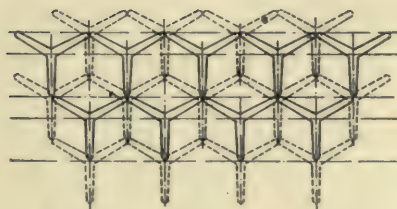


FIG. 106.

also made from a suitable heat and acid-resisting substance, the object of this filling material being to facilitate the contact between fumes and liquor, and in this manner to effect the absorption, reaction, mixing, or cooling. It will be therefore readily understood that the more efficient this filling material can be made in proportion to its bulk, the smaller the tower can be in order to carry out the same amount of work as a larger tower with a comparatively less efficient filling material. Numerous attempts have been made to provide a filling material which, besides being efficient, would occupy a minimum amount of space, and in this way

reduce the size, and consequently the cost, of such a tower as much as possible. It will be obvious that the cubic space representing the displacement of the actual material of the filling elements may be regarded as waste space in the tower, and that the ideal filling material is that which affords a large amount of so-called free space and a large surface per cubic unit, combined with cheapness of manufacture and efficiency in action. As the synthetic processes for the manufacture of acids and the like are rapidly developing, the demand for towers which, as above stated, form an all-important factor of these processes, and consequently for a suitable filling material, is also largely increasing.

Figs. 104 and 105 illustrate in elevation and plan view respectively one form of the filling element, and Fig. 106 illustrates in plan view one method of disposing the elements in a tower.

The first important point for consideration is the cheapness of manufacture, and this is solved by giving the filling element a shape which will permit of its being formed by pressing the plastic material of which it is composed through an appropriately shaped die by means of a suitable press, and then, either by hand or otherwise, cut off to a convenient length as it emerges from the die, in a manner somewhat similar to that adopted in the manufacture of earthenware pipes, etc. The filling element consists of three or more wings or arms which radiate from a centre, and may be straight, or preferably twisted or helical as shown. In some cases the central portion may be enlarged, and a hole formed longitudinally from end to end. A filling element of this construction affords a large free area and an extensive surface. For example, a filling element as is shown with three twisted wings of a diameter of 15 centimetres, and a height of 15 centimetres, has an effective surface of approximately 1,100 square centimetres. This surface may be increased considerably by corrugating it. The total cubical contents of such a filling element approximates to 420 cubic centimetres. As the cubic metre will contain approximately 480 of such elements, when the same are arranged as shown in Figure 106, for example, it will be seen that only 20% of this space is taken up by the solid parts of these elements, thus leaving as much as 80% of free space. It will be also seen that the effective surface will amount to approximately 53 square metres per cubic metre of filling elements, or, if the surfaces are corrugated as described above, the total effective surface will amount to approximately 70 square metres per cubic metre occupied. It will be therefore obvious that by employing a filling material similar to that described, it will be possible to reduce the size of the tower considerably without reducing the efficiency. The efficiency is increased by employing the twisted or helical shape of wings α by which the fumes

or gases passing through the tower are caused to assume a whirling motion by which the fumes or gases present continually changing surfaces to the filling material, which naturally improves the reaction taking place between the gases or fumes and the liquor. If desired the helical wings of the elements may be made right and left-handed, and may be disposed alternately in the tower, that is to say, first a row or layer of right-handed elements, and then a row or layer of left-handed elements, for example.

Nielsen (*Chemical Engineering*, Vol. V., p. 346) in describing his filling material with special reference to its use in the Gaillard tower, observes that in the Gaillard system of concentration the sulphuric acid to be concentrated is caused to fall in a fine shower or spray down an empty tower, which may be 30 or 40 feet high, and in its downward

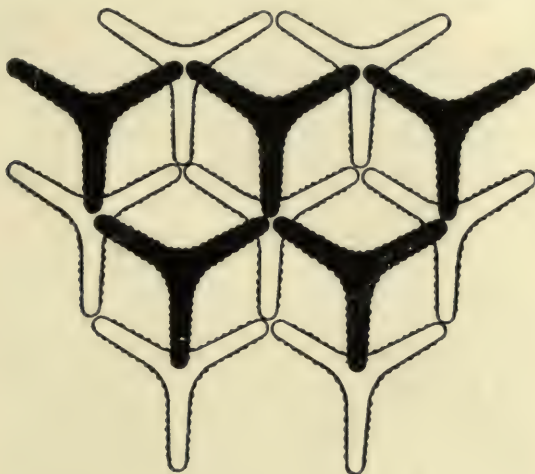


FIG. 107.

course it meets an ascending current of hot combustion gases from a suitable producer or coke fire. The process is simple to the verge of primitiveness. In this system, obviously the time during which any acid particle is exposed to the concentrating effect of the hot gases is the period occupied by its descent in the tower. This cannot exceed three, or at most four seconds, so to secure efficiency the gas temperature must be high, and, as is well known, the employment of these high temperatures has many disadvantages, causing quick destruction of plant, and loss through formation of acid fog, etc. If the acid particle could be delayed in its fall, the same evaporative effect could be secured by employing a larger volume of gas at a lower temperature.

This longer contact period or retarded fall of the acid particle can

only be secured by filling the tower with suitable filling material, but a filling or packing to meet the working conditions must be one of low first cost, and of great durability; it must have large contact surface for the acid film, and great free space for the hot gases; it must have no corners or perforations to be blocked up by dust from the gases, and capillary action, causing channelling, must be avoided by avoiding surfaces more or less horizontal. Lastly, the ascending gases or fumes must meet with the least possible resistance, so as to maintain the draught. "Propeller" filling is stated to meet these requirements; it will also be found to adapt itself to any form of tower—round, square, or oblong (see Fig. 107.) Such towers packed with propellers of any of the standard sizes, still have almost 80% of space free for the gases, the tower is not filled with masses of inactive stoneware, but the space is scientifically

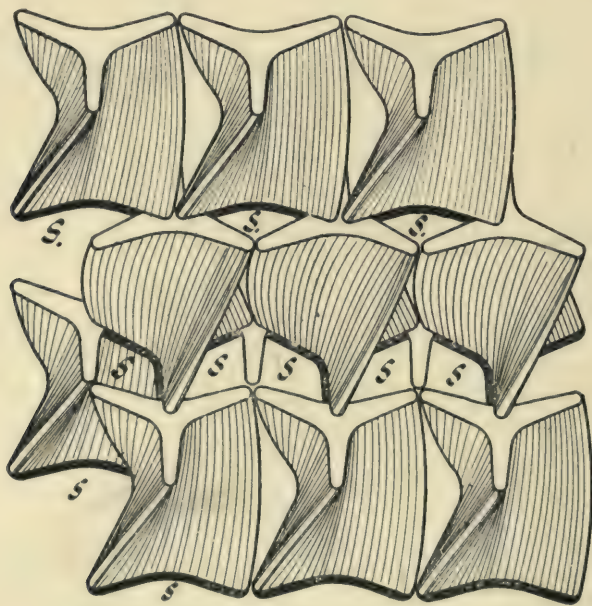


FIG. 108

divided up by a system of curved planes, designed to direct the gases against wet surfaces, these being at the same time of such a form as will give the descending acid the longest contact period, but will not permit dust to gather, should it be present in the gas. The area for gases in "propeller" packed towers is practically the same from top to bottom, so that a minimum resistance to the flow of the gas is offered. The whirling of the gases produced varies in direction at each layer, and is due to the suitable curvature of the propeller blades (see Fig. 108.)

In this way only can efficient contact be secured between liquid and gas without loss of gas "pressure."

Gaillard towers packed with filling material work with peculiar regularity owing to the recuperating property of the filling. If producer gas is made use of this effect is not so obvious as when a coke fire is used as the source of heat. In the latter case, each time fresh fuel is introduced there is (in the case of an empty tower) a sudden drop of temperature, followed by a sudden rise of temperature, and in neither case can the acid supply be correspondingly adjusted. The result is that at the low temperature there is a loss of evaporative effect and at the high temperature a loss of acid, owing to the formation of "fog." If, however, the tower is filled—the material in large towers may weigh over 100 tons—the hot filling acts as a splendid equalizer and recuperator, and temporary sudden changes in gas temperatures can produce no bad effects, and this steady working at moderate temperatures is claimed to effect distinct economy in fuel. Heat losses through radiation and conductivity are practically eliminated, and so also the acid losses due to over-heating. With the ordinary empty Gaillard tower a final coke-filled tower is frequently installed. With a properly filled tower this is not necessary, and so a serious drawback to the Gaillard system is avoided.

Nielsen observes that a point well worthy of taking into consideration in the design of filled Gaillard towers is the possible reduction in size owing to the highly intensified contact, or heat exchange, between the acid and the hot combustion gases, due to the propeller filling. His own experience with absorption towers and gas scrubbers points in this direction.

"Obsidianite" Packing.—The qualities of "Obsidianite," made by Messrs. Charles Davison and Co., Ltd., of Ewloe, near Chester, as an acidproof and fireproof material, were well-known before the war to many chemical engineers, but the enormous demands for highly concentrated sulphuric acid for the manufacture of high explosives has brought this excellent material into much more general use. Germany had supplied the materials for Kessler concentrators, and France, which had supplied the volcanic stone for the Gaillard type of plant, needed for her own use all and more than all of that material which she produced. The result has been the wide-spread adoption of "Obsidianite" for the whole range of purposes in connection with Glover and Gay Lussac towers, concentrators of the cascade and Gaillard types, and with furnaces for burning sulphur, pyrites, spent oxide, zinc blendes, etc. The makers claim that Obsidianite will not only

outlast the foreign acidproof materials in the matter of its resistance to the action of acids, hot or cold, concentrated and dilute, but will also stand a high temperature, which they will not.

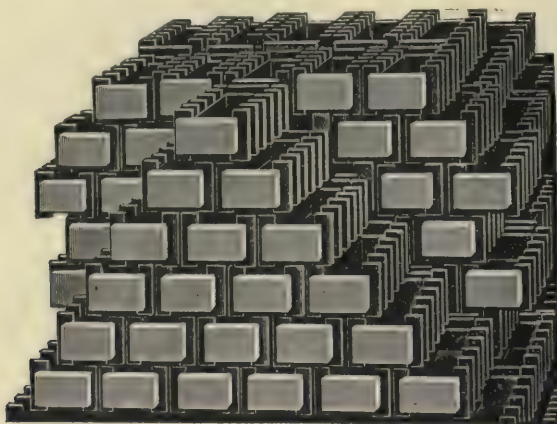


FIG. 109.

Fig. 109 shows the Obsidianite patent packers, and indicates the method of packing; the use of this material gives great flexibility and control in working the tower.



FIG. 110.

Fig. 110 shows the Obsidianite arches, grids, and ring packing for Glover towers, each block being fitted and numbered for re-erection.

Obsidianite Gaillard Towers.—Before the war it was almost invariably the practice to construct Gaillard towers of volvic lava, as it was believed that nothing else would make close joints, but before the war, in a large explosive works in South Africa, a large Gaillard

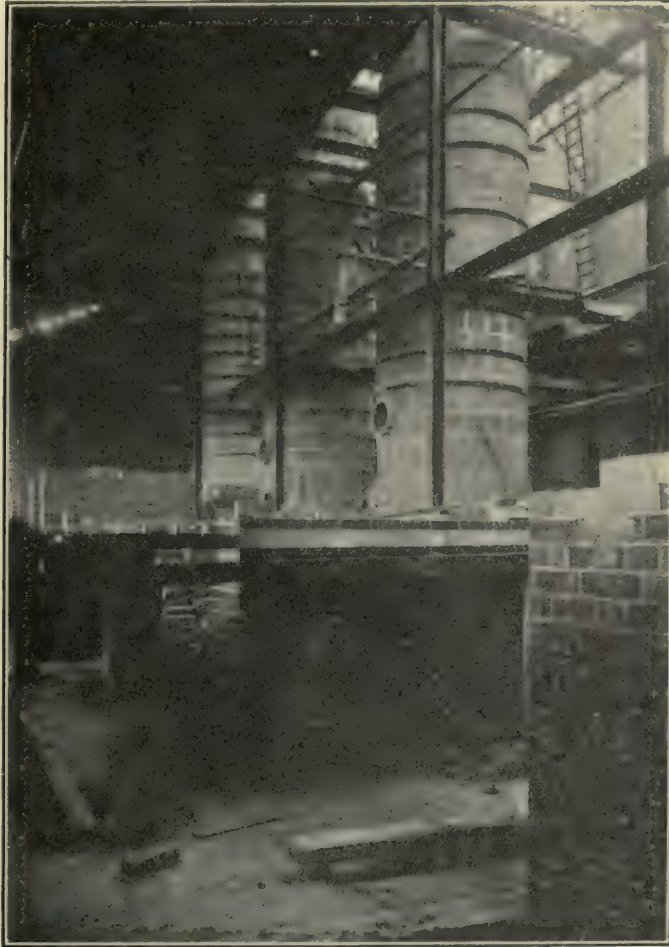


FIG. III.

tower had been constructed of Obsidianite, the walls being built up of large thin slabs of Obsidianite covering each other at the joints in layers of varying thickness. This construction proved very successful; after a short time there was no leakage, and a life was secured

of two or even three times that of a tower built of volvic lava. Since 1914 over seventy Gaillard towers in Obsidianite have been supplied by the makers to the French Government, and in the United Kingdom a large number of such towers were erected in Government and other works.

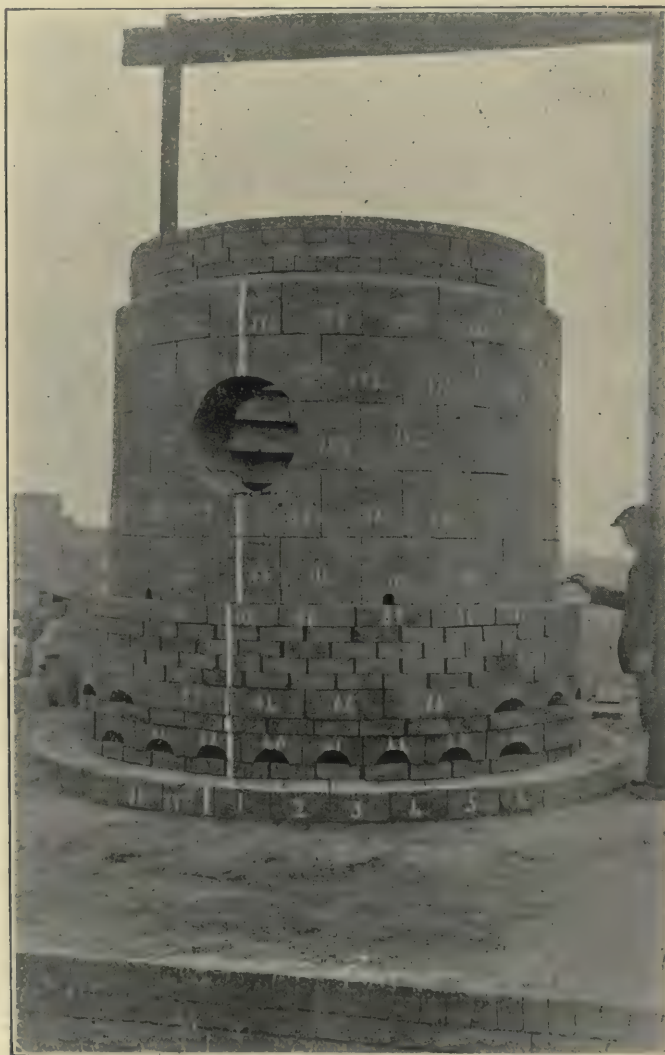


FIG. 112.

Cascade Concentrators.—The causes which led to so many manufacturers abandoning the cascade type of concentrator have to a very great extent been obviated and overcome by

the long and persistent experiments and improvements of the Thermal Syndicate, Ltd., who, in conjunction with Messrs. Davison and Co., have succeeded in producing a really satisfactory and reliable plant. Improvement in the efficiency of this cascade type of concentrator,

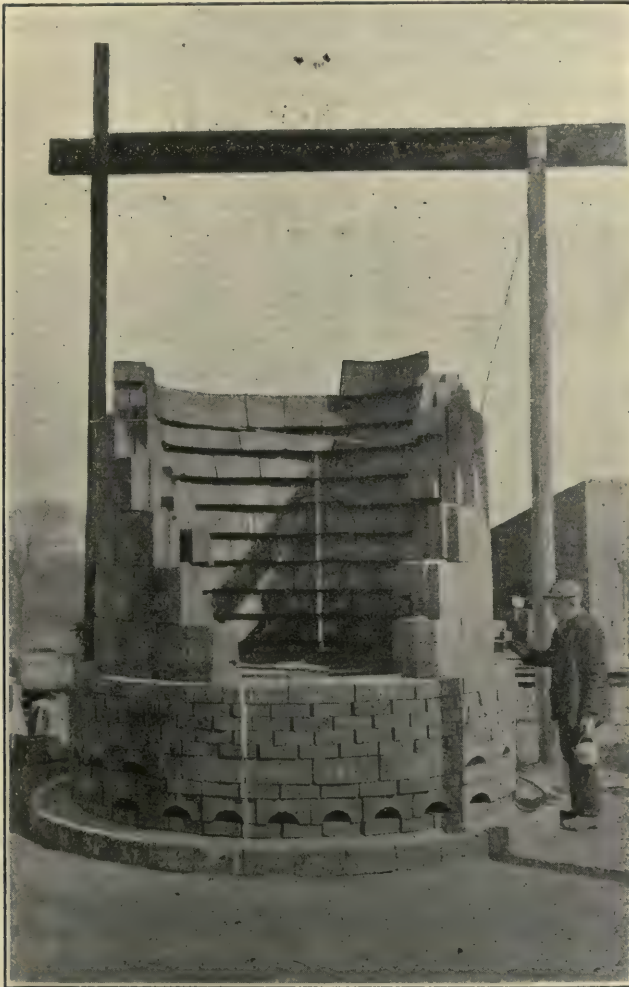


FIG. 113.

and the lengthening of its life, is aimed at mainly by an improved method of distributing and regulating the fire gases in the combustion chamber. The employment of gas as the method of combustion would probably be the ideal arrangement, and a perforated roof to the fire flue, in order to allow better distribution of the flame,

would probably improve matters. But in the opinion of the makers, to those who can afford the space and the capital expenditure, a battery of Gaillard tower concentrators, with recuperators, provides a plant which is efficient, simple in working and in management, the most economical from every point of view, and the most durable type for practical use. The author is permitted to publish (Fig. 111) an exclusive official photograph of such a battery in course of erection for the Ministry of Munitions. This is entirely erected in Obsidianite fire and acid-proof material, and is one of a large number of similar batteries, ranging from four to six in number, already erected and erecting in Government and controlled works in this country. The author is informed that some of these towers have been working at high pressure for a considerable time and are giving the best results. Hitherto Gaillard towers have almost invariably been erected in large blocks of volvic lava, and it is gratifying to find that at a time when volvic lava is not available, British acid manufacturers have been able to find at home in Obsidianite not only a substitute, but an even more satisfactory because much more durable material for their erection. In Figs. 112 and 113 are shown another type of Gaillard concentrator erected in Messrs. Davison and Co.'s patent overhanging and overlapping Obsidianite blocks, which are impenetrable by acid even at the joints, with which the acid is quite unable to come into contact. Towers of this type have been erected in some of the largest works in the North of England.

Notes on Cascade Concentrators.—The following official notes on cascade concentrators (including the recovery of waste acid from picric and T.N.T.) have been issued to manufacturers.

All concentrating plants for R.O.V. should be supplied with the hottest and strongest acid which it is possible to obtain by concentration in lead pans.

The most economical and efficient method of concentration of chamber or de-arsenicated sulphuric acid up to 140° Tw. is in open lead pans working on the continuous flow system.

It is false economy to use silica ware for warming up and concentrating acid to a stage which can be done far more efficiently in much cheaper lead pans.

There is much to be said in favour of the view that the wear on a concentrating plant is better gauged by the hours that it is in operation rather than by the tonnage of its output. At least, it is certain that, within wide limits, an increase in output per hour does not at all

correspond to a proportionate increase in wear. An increased output per hour means a saving in labour per ton.

It is extremely wasteful, both from the point of view of capital and labour costs, to burn poor coal or to work a plant with a poor draught.

Where new cascade concentrators are to be erected the open lead pans can be placed on the flue behind the basin plant, and thus the waste heat from the basin cascade can be utilised and hot acid at 140° Tw. fed into the covered basins.

Where weak acid of 110° Tw. or lower strengths is to be concentrated the waste heat will probably be insufficient, and either auxiliary firing of the lead pans or a separately fired lead pan set should be provided.

The flat open silica trays are only really preheaters, and do not allow sufficient time-contact for much concentration to take place.

No covered basin plant should be fed with acid below 140° Tw., and where basin cascade plants are already in existence lead pans should be erected without delay as a separate unit, separately fired, to bring the acid up to this strength.

From 40 to 50% increase in production can be obtained by feeding cascade concentrators with 140° Tw. acid as compared with the plant fed with 110° Tw. acid.

This remark applies equally to Kessler and Gaillard tower concentrators.

Weak acid attacks the ferro-silicon iron basins more readily than strong acid, consequently the top 12 basins in a covered run of 25 should be of silica ware if contamination with iron is undesirable.

The installation of a cast-iron pot at the bottom of the cascade basins increases production considerably; the pot should be flat and shallow, and there should not be more than six inches of acid in it, as otherwise silting up with ferric sulphate occurs and efficiency is reduced. The cast-iron pot should have a separate fume pipe, and, as the gases evolved are very hot and corrosive, special earthenware or ferro-silicon pipes are desirable. The gases from the cast-iron pot, if separately condensed, give a fairly strong condensed acid, and the strength of this condensate would give a good indication of firing and other working conditions.

The pot should be covered with stone or earthenware slabs, or ferro-silicon iron covers. The cooler should be of cast-iron of large size, the hot acid should be delivered well down into the bulk of acid in the cooler, which should be provided with a large lead pipe water coil and suitable tapping out hole at the bottom for letting off the sulphate of iron mud.

The cooling coil of lead pipes should be right up against the side of the cooler in order to be as far removed from the hot acid inlet as possible. The length of the water coil must be sufficient to bring about the necessary cooling of the acid. Many works engineers fail to realise the importance of this.

The acid entering the iron pot should not be below 158° Tw., and a higher figure of, say, 160° Tw. or 162° Tw. is desirable, otherwise excessive corrosion of the iron pot occurs.

Where an iron pot is installed, 20 covered basins in series are sufficient if fed with hot 140° Tw. acid.

DRAUGHT ON THE CASCADE FLUE.—As the sole aim in concentrating weak acid is to remove water, it is obvious that the water vapour should be removed as rapidly as possible from this flue. It is therefore essential to have a good draught, in fact, the pressure in this flue should be decidedly lower than in the furnace flue, so that leakage, if any, should be from the furnace flue to the acid flue. Such a result can be obtained by having scrubbers of sufficient area with fairly open packing, ample area of connecting pipes, and strong suction on the end of the scrubbers. Natural draught from a good chimney and a fairly long flame coal give the most even distribution of heat up the cascade run. Intense local heating is to be avoided, as this only leads to dissociation of the acid, a large quantity of distillate, and to excessive breakage of the basins. Condensers for cascade concentrators should be packed with a fairly open packing, otherwise installation of fans for draughting purposes may be necessary. The asbestos rings are not gas-tight when once heated, and back pressure on the acid fume flue considerably retards production.

A steam injector at the inlet to the condenser assists the draught, and also assists condensation of the acid fume, but if the condensers are worked cold some dilution of the distillate may occur. This dilution can be largely avoided by working the condensers hot, or by putting the steam injector before the final wash tower where a series of tower condensers are in use.

It is good practice to condense the gases from the iron pot in a separate condenser. The condensed distillate from the iron pot should be allowed to flow continuously through a Twaddell jar so that the firing and flow of acid can be controlled. This is a simple but effective means, and is invaluable when high strength acid is being made. Naturally the condenser must be so designed that little acid stands in it, or otherwise the indications of change of strength of distillate, and consequently of strength of concentrated acid being produced, follow too slowly.

RECOVERY OF WASTE ACID FROM T.N.T. MANUFACTURE IN CASCADE CONCENTRATORS.—The previous notes as to lead pans apply.

The pans should be installed as primary concentrators behind the basin cascade to bring the acid up to 140°-144° Twaddell.

If the waste heat is not sufficient to bring the acid up to required strength in the lead pans, it is most necessary to have separately fired supplementary lead pans.

Preliminary denitration of the waste acid prolongs the life of the lead pans, but where the feed acid passes through a recuperator tower, fed by the hot gases from an iron finishing pot, other preliminary denitration is unnecessary.

Denitration by means of the vapour from the iron pan has advantages, but it does away with the possibility of using the strength of the distillate from the iron pan to control the working of the plant, a procedure which is very useful.

Denitration with superheated steam dilutes the acid less than when ordinary wet steam is used.

A covered basin cascade will only with difficulty produce a good yield of 95% acid from this class of waste acid, consequently an iron finishing pot to decompose completely the nitro bodies, and to procure a 96% acid, is highly desirable.

The previous notes as to iron pots apply.

One method is to take the hot gases from the iron pot through a recuperator fed with feed acid for the cascade. Thus partial condensation of the hot fume is obtained, and denitration of the feed acid is procured without great dilution.

The alternative method of separate denitration with superheated steam, with separate condensation of the iron pot fumes, and regulation of the working of the plant by the strength of distillate produced from the iron pot, is quite good practice.

Condensers of stone or earthenware packed with rings have given quite satisfactory results; a steam injector at the inlet of the condensers assists draught, and helps to keep the condensers hot.

It is very essential that the condensers for the fume from this class of waste acid should be worked hot, otherwise blockage occurs from decomposition of dinitro and trinitro bodies.

The recovery of a 96% to be used for further nitrating operations is a very important factor in the saving of oleum, and should be attained in every case.

RECOVERY OF WASTE ACID FROM PICRIC MANUFACTURE.—This should be done in two stages:—

(1) Concentration up to a point where the maximum amount of

picric acid can be removed without decomposition. This has been found to be from 127° – 135° Tw.

This can only be economically done in a cascade system of open lead pans with continuous flow, the lead pan area required being from 20 to 25 sq. ft. per ton of 130° Tw. acid produced per day. This is calculated on working with continuous flow and firing, and with about 9 inches of acid in the pans when the feed acid is of about 70° Tw. The acid running off should be partially cooled until it is just about to deposit crystals, and then run into large settling tanks, in which the picric acid is allowed to crystallise out slowly. These settling tanks can be either lead-lined or of stone. Where lead-lined settling tanks are in use the picric acid should be scooped out and taken to the washing plant in the usual way. Where stone tanks are in use there is no objection to washing in the stone tank, if desired, after running off the cold acid.

The partially concentrated acid should on no account be used for the manufacture of nitric acid, as it still contains some picric acid which might give trouble in the retort.

(2) Further concentration of the 130° Tw. acid, from which the picric acid has been crystallised out, in basin cascade concentrators up to 95% strength.

This can quite well be done in the ordinary silica basin concentrators with silica tray preheaters, when a clean acid eminently suitable for further sulphonation of phenol can be obtained.

In works where efficient chemical supervision could be maintained, an iron finishing pot could be used as mentioned previously, but it is essential that the acid entering the iron pot should be not below 92% strength, otherwise contamination with sulphate of iron might result, with a consequent rise in the ash content of future batches of picric acid.

Condensers for fume should be of stone or earthenware with open packing such as rings. Steam injection at the inlet to condensers and working of the condensers warm assist in preventing blockage.

The capital cost of the above method per ton of 95% acid produced per day is considerably less than that necessary for attempting to do the first concentration in silica cascade plants; whilst the attempt to recover a 95% acid in one operation with all the picric acid and sulphonated bodies in solution is impracticable.

Obsidianite Cascade Concentrator Plant.—Figs. 114 and 115 are reproductions from exclusive photographs, which the author is permitted to publish by the courtesy of Messrs. Charles Davison and Co. and the British Admiralty, of what is believed to be the largest

cascade concentrator plant in the world. The plant, which was made by Messrs. Davison and Co. for the Admiralty, is of Obsidianite fire

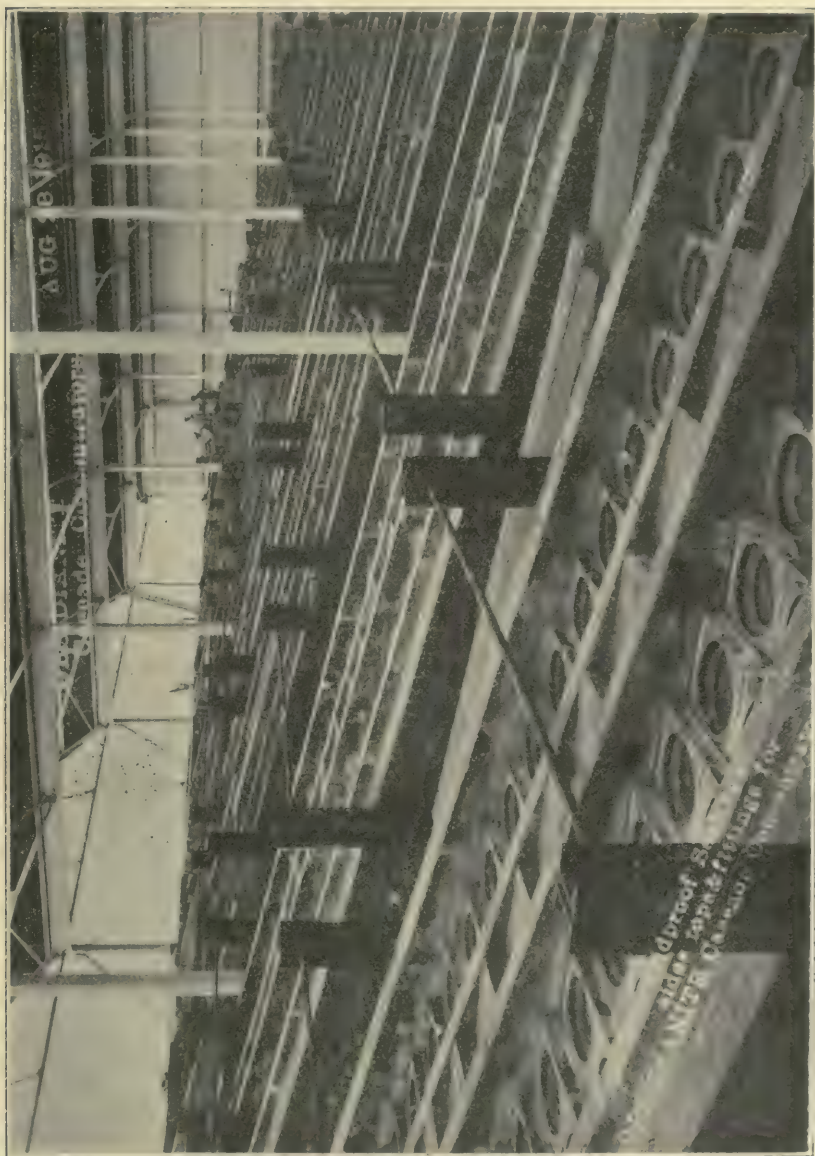


FIG. 114.

and acid-proof material; in the photographs the Obsidianite covers are seen removed to show the interior arrangement.

The old cascade concentrators had a very small output of acid of moderate concentration, but such a plant as that here illustrated



FIG. 115.

gives a very large and constant output of the most highly concentrated acid (98% H_2SO_4). This means an intense temperature and

intensive acid conditions. In making cascade concentrators of the old slow type, any ordinary acid-resisting materials would answer the purpose for quite a considerable time. But when 98% H_2SO_4 had to be produced day and night in the greatest possible quantity, the heat required was very great, the seatings melted away, the sides fell in, the covers shaled off under the attack of the condensing acid, and fell into the basins, which they destroyed. Consequently the plant was frequently stopped, the acid lost, and the cascade concentrator condemned as inefficient. The combination of "Vitreosil" basins and trays with Obsidianite fire and acid-proof seatings, sides, and covers has, however, completely overcome these difficulties. In order to stand the great heat of the fire gases necessary for 98% concentration, the lower seatings are made with closed bottoms as the fire gases lose first intensity, and by open supports as soon as the heat is sufficiently moderated not to crack the basins when brought into direct contact with them. Finally, beyond the covered-in basins there are arranged double rows of shallow "Vitreosil" trays seated over the flues on Obsidianite lock-jointed and self-locking tray supports, which arrangement economically allows of the acid being gradually heated up to the necessary temperature before the actual concentration commences.

In some notes on the working of sulphuric acid concentration plant in 1915, the Chief Inspector of Alkali Works observed that the working of the cascade system of stepped vessels was the greatest cause of trouble through the heavy demands for concentrated acid for explosives manufacture. For the efficient and satisfactory operation of the cascade type of concentrating plant it is essential that (1) a greater draught should be given to the flue leading the evaporated gases and vapours towards the condensers than is given to the fire flue leading the fire gases to the chimney; (2) excessive local intense heating of any section of the cascade should be avoided; and (3) adequate means for condensing the acid vapours be provided. Sufficient draught on the fume flue ensures the proper removal of vapours of evaporation; it thus increases the amount of work done by the plant, and at the same time prevents the objectionable acid fumes being drawn into the fire flue to pass thence by way of the chimney into the air. Intense local heating leads to badly balanced evaporation, to dissociation of the acid, with a consequent undue formation of fume, to violent ebullition in some of the vessels, leading to breakages, entailing further formation of fume accompanied by general deterioration of the plant. Adequate condensers are necessary in the interests of the public, and are practicable on all such plants. From the experience already gained in the past, the tables given have been compiled for information of those who have to do with the erection and operation of these plants:—

CASCADE PLANT AND CONDENSERS.

Basins of Silica, Ironac, Narki, and Tantiron, with Preheaters.

| Acid Feed Strength. | Number of Basins. | Output Tons Acid per 24 hours. (160—160° Tw.) | Water and Acid evaporated per 24 hours. Tons per 100 Basins. | | | Packing in Condenser. | | | |
|---------------------|-------------------|---|--|-------------|--------------|-----------------------|---------------------------|------------------------------|---------------------------|
| | | | Loss of Strong Acid assumed to be | | | Per Basin. | | Per ton of Strong Acid made. | |
| | | | Nil. | 5 per cent. | 15 per cent. | Volume Cubic Feet. | Sectional Area Square Ft. | Volume Cubic Feet. | Sectional Area Square Ft. |
| 105° Tw. | 727 | 61.5 (=1.69 cwt per basin) | 4.5 | 5.1 | 6.4 | 6.5 | 1.6 | 77 | 18 |
| 123° Tw. | 1,440 | 112 (=1.55 cwt. per basin) | 3.1 | 3.6 | 4.7 | 8.2 | .7 | 105 | 10 |
| 136° Tw. | 40 | 4 (=2.0 cwt. per basin) | 2.7 | 3.3 | 4.6 | 6.2 | 1.2 | 62 | 12 |
| 147° Tw. | 100 | 15 (=3.0 cwt. per basin) | 2.9 | 3.8 | 5.5 | 7.5 | — | 50 | — |
| 120° Tw. | 40 | 2.5 (=1.25 cwt. per basin) | 2.5 | 2.9 | 3.8 | 5.0 | — | 76 | — |

Cascade Plant and Condensers with Preheaters.

| Acid Feed Strength. | Description of Plant. | Output of Acid. | | Nature. | Packing in Condenser. | | | |
|---------------------|---|--------------------------|---------------|-------------------|-----------------------|-----------------------------|------------------------------|-----------------------------|
| | | Tons per 24 Hours. | Strength °Tw. | | Per Basin. | | Per Ton of Strong Acid made. | |
| | | | | | Volume Cubic Feet. | Sectional Area Square Feet. | Volume Cubic Feet. | Sectional Area Square Feet. |
| 110° Tw. | 3 Tantiron Pans and Basins. | 5 | 168 | Coke | — | — | 43 | 7 |
| 120° Tw. | 12 Tantiron dishes 80 (3 ft. diameter). | 12 | 166 | Bricks and Tiles. | — | — | 30 | — |
| 126° Tw. | Silica Basins & 2 iron “pots” 8' × 3'). | 14 | 168 | Rings | — | — | 15 | — |
| 135° Tw. | “Webb” plant—30 Vessels. | 4.5 (3 cwt. per Vessel). | 168 | Coke and Bricks. | 14.5 | 15.1 | 96 | 101 |

— HAUGHTON'S 'IRONAC' CASCADE CONCENTRATION PLANT. —

INCORPORATING THE HAUGHTON-TUNCAY PATENTS.

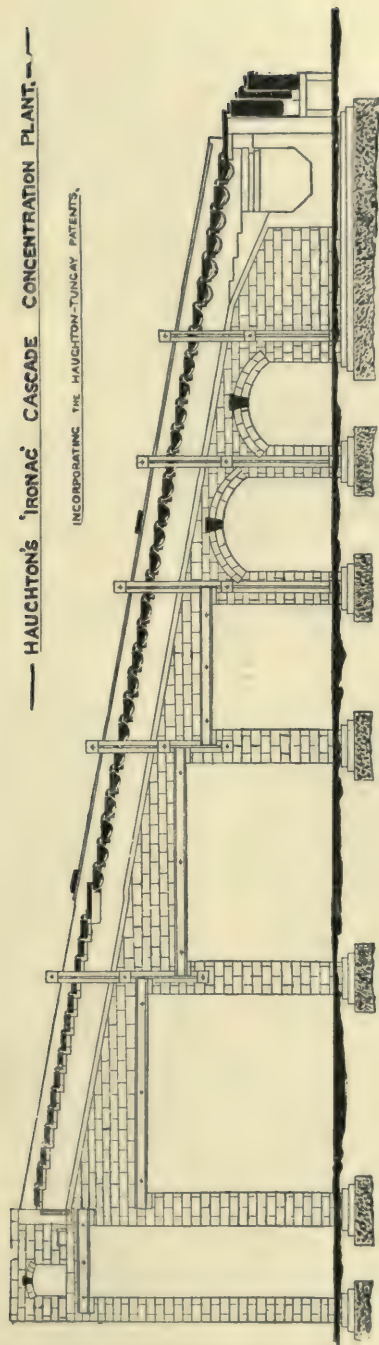


FIG. 116.

Haughton's "Ironac" Cascade Concentration Plant.—Fig. 116 shows a cascade concentration plant in "Ironac" metal, by Messrs. Haughton's Patent Metallic Packing Co., Ltd., of 30, St. Mary at Hill, London, E.C. The plant as illustrated shows the general arrangement of an improved cascade plant constructed in series, and having a capacity of either 5, 10, or 20 tons of concentrated acid per 24 hours.

The whole of the parts in contact with acid are made according to the Haughton-Tungay Patents. The pre-heating of the weak acid, which is usually fed into the plant cold, at a density of say 108° Tw.- 110° Tw., is dealt with in a series of rectangular trays with outlet lips for



FIG. 117.

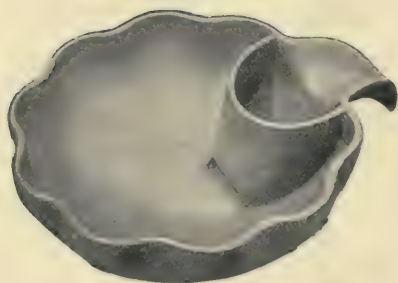


FIG. 118.



FIG. 118

pre-heating the acid, arranged alternately right and left hand in such a way that the acid has a long traverse over the flue gases until it is carried through a leading-out pipe into cascade basins for final concentration. The pre-heating trays (Fig. 117) are arranged uncovered and are always open to inspection, no objectionable fumes being given off this section of the plant.

The concentration proper takes place in the cascade basins, (Fig. 118) which although of the standard "Benker" size, are modified in design, having corrugated upper edges to allow of a free expansion and contraction whilst under heat, and to prevent any overflow or leakage through frothing of the acids during concentration. All the cascade

HAUGHTON'S 'IRONAC' CASCADE CONCENTRATION PLANT.

INCORPORATING THE HAUGHTON-TUNNEY PATENTS

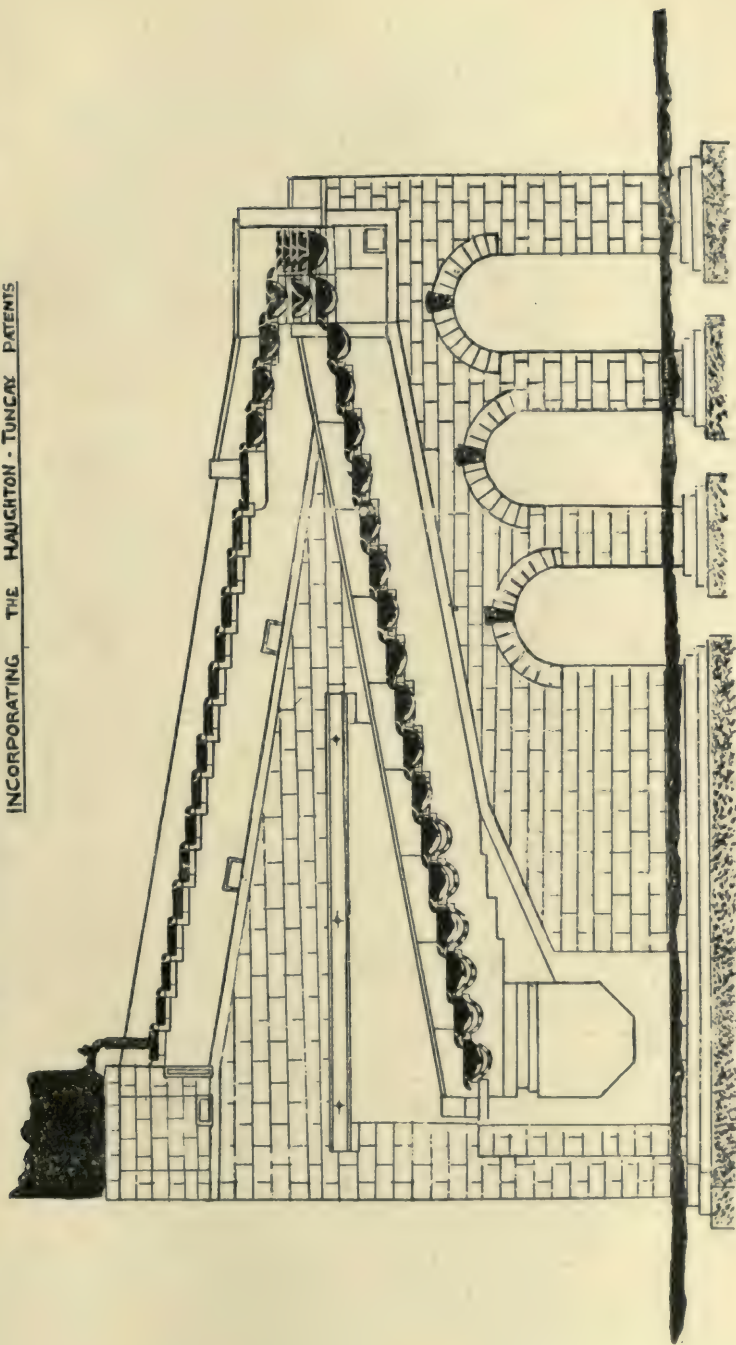


FIG. 119

basins are covered in such a way that any acid fumes distilled off can be carried away through a series of fume pipes to condensers or scrubbers. When concentrated up to 168° Tw. the strong acid is carried off through beaker pipes into a system of pot coolers which are surrounded by cold water circulation. The pot coolers are very efficient owing to high heat conductivity of Ironac.

(Fig. 117) shows one of the pre-heating trays showing the side outlets, which are arranged alternately right and left hand. Fig. 118 is a view of the Haughton-Tungay cascade basin, which is fitted with projecting curtain in such a manner that only the strong acid which descends to the lower part of the cascade basin is allowed to flow out from the outlet or overflow. Thus it will be seen that all the acid must be brought in contact with the hot gases, and can only escape from each successive basin when it has acquired the higher density and gravitates to the lower part.

Fig. 119 shows a further modification of the Haughton-Tungay system of "Ironac" cascade concentration plant. It has been found that some of these plants occupy a considerable amount of floor space, and for positions where floor space is limited it has been found advisable to arrange the plant as shown in this illustration. A number of plants have been laid out on this design, and it will be seen that the fire flues as well as the cascade itself are turned back midway along the run of the cascade, in such a manner that the entire plant only occupies about one-half the usual length of floor space which an ordinary run of cascade plant would occupy. This system of cascade plant can be adapted either for the concentration of chamber acid or the recovery or re-concentration of waste acids which have been used in connection with explosives, nitration processes, or other industries. All the vessels and parts which contain acid, are made of Ironac metal.

Fig. 120 shows the latest design of Ironac cascade concentration plant adapted for the reconcentration of waste acids from nitration plants, in connection with explosives, guncotton, T.N.T., anilines, and other processes. This plant embodies the Haughton-Tungay improvements in respect of pre-heating trays and cascade basins, which are made of Ironac, the latter being fitted with dividing curtain having bottom outlet so arranged that each cascade basin will only deliver the concentrated acid into the next stage.

The waste acid from which it is desired to rectify the sulphuric, has first to be denitrated by means of denitration towers, either worked by steam or superheated gases, and is then delivered at the top of the cascade for primary treating in the pre-heating trays. The cascade basins, which are enclosed and from which H_2SO_4 gases are certain

HAUGHTON'S IRONAC' CASCADE CONCENTRATION PLANT.

FOR RECONCENTRATING WASTE ACIDS
10 TON PLANT
INCORPORATING THE HAUGHTON-TUNGAY PATENTS.

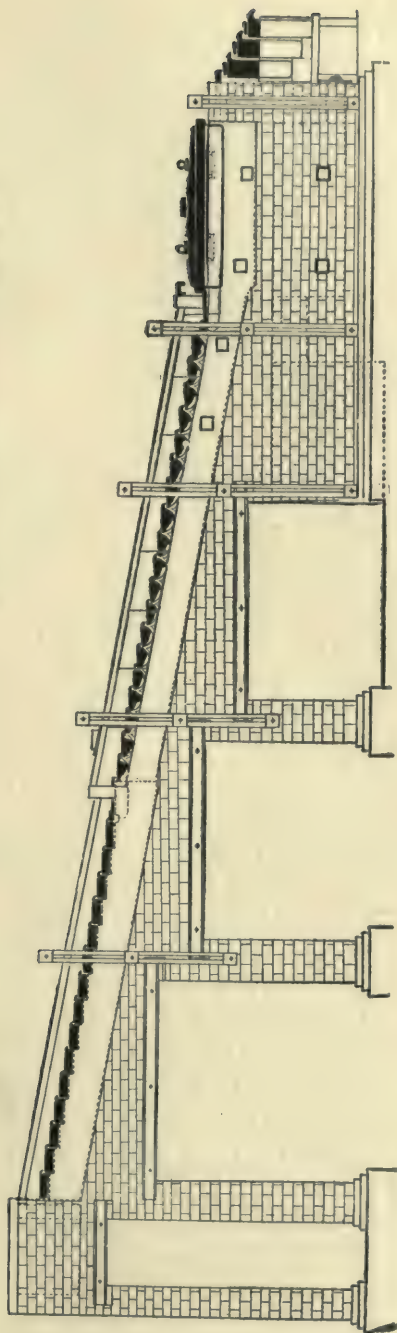


FIG. 120.

to be distilled off to some extent, are so arranged that by a system of fume pipes the gases can be led away to scrubber condensers.

As soon as the acid is concentrated to such a degree that it no longer corrodes cast iron, it is conducted through a beaker pipe into a large shallow rectangular cast-iron pan fitted with removable cover. It is in this pan that the final concentration up to 96½% or 97% sulphuric acid takes place. The cover of the cast-iron pan or still is also fitted with outlet for fume pipe, so that any sulphurous gas distilled off can be led away to scrubbers for recovery. The author is informed by the makers that it is found by this means that waste acid requiring re-concentration can be more effectively treated than by relying upon a system of cascade basins alone.

After treatment in the cast-iron still, the rectified acid is led through a beaker pipe to a system of pot coolers which are water cooled. The cover of the pan or still is arranged to be removable, so that this may from time to time be opened and cleared of any solid matter or precipitate which may be thrown down from the waste acid when reaching the higher concentration.

Fig. 120 shows the general arrangement of a plant having a capacity of 10 tons of concentrated sulphuric acid per 24 hours.

Fig. 121 shows a plant consisting of a system of shallow pans of 36 inches diameter made entirely of Ironac. Each pan has a socketted inlet and an overflow lip or outlet arranged at a suitable level for drawing off the acid in course of concentration, and each pan is also furnished with a separate removable cover to which is attached a fume pipe for drawing off the acid fumes. A series of these shallow covered pans are arranged in cascade fashion upon a suitable fire-brick seating as shown in the illustration, and in this manner they form a cascade concentration plant of a visible type, that is to say, all the concentration vessels are always visible and open to view, so that any defect in working can at once be located and remedied. The fume pipes leading from each of the covers are connected to a main water-jacketted pipe for carrying off the waste gases to scrubber or condenser.

The author is informed that the working of this plant has been found very successful, and the plant as illustrated has an output of 10 tons of concentrated sulphuric acid of 168° Tw. per 24 hours, the acid at the inlet being 106° to 110° Tw. One of the special advantages of these pans of large diameter is that a very greatly increased heating surface is in constant contact with the hot gases of the furnace. It will also be observed that the erection of the plant requires a minimum amount of brick-work, and occupies considerably less ground space than the older types of cascade plants using small basins.

HAUGHTON'S
VISIBLE TYPE - 'IRONAC' CASCADE CONCENTRATION PLANT.
10 TON PLANT.

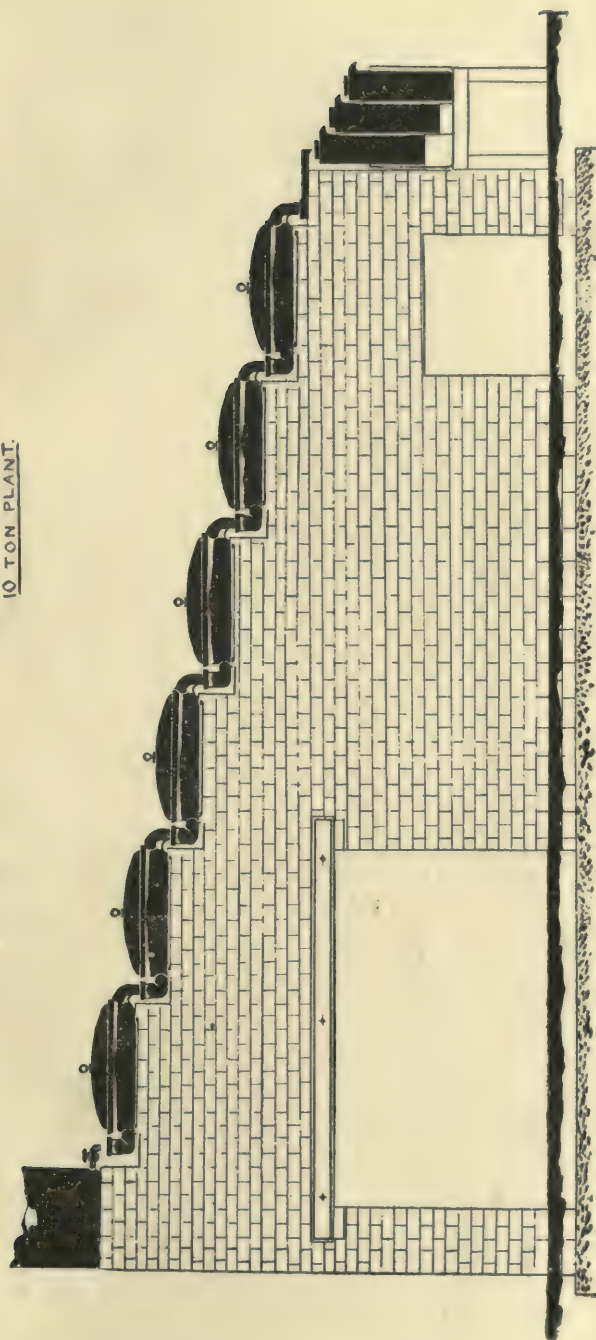


FIG. 121.

Zanner's Concentration Plant.—Figs. 122, 122A and 122B show the plant of Zanner, for the high concentration of sulphuric acid (British Patent No. 19,481 of 1913). Zanner observes that the high concentration of sulphuric acid by direct heating was formerly effected chiefly in glass retorts, platinum apparatus, dishes and cups. The glass retorts gradually disappeared from practical use, the principal reason being probably, that by their means only intermittent working was possible. The platinum appliances had to contend with the extraordinary rise in the price of platinum in recent times, and have consequently almost completely disappeared from technical working. Zanner continues:—"The concentration in dishes as practised, for instance, by Negrier or Benker, and the concentration in cups (Webb) can, owing to the one-sided heating, no longer compete with the modern hot-air processes, extremely economical in the consumption of fuel, such as those introduced by Kessler and Gaillard. On the other hand the hot air processes require complicated plant and large quantities of special fireproof materials (volvic lava), which are expensive. My object was to render high concentration as cheap and simple as possible. The first result of my endeavours in this direction was the Patent No. 7999 of 1907, according to which the heat of the gases of the roasting kiln was utilized for raising the chamber acid (50 to 53° Bé.) or Glover acid (60° Bé.) to 66° Bé. by means of pans mounted freely in the flue for the roasting kiln gases situated between the kiln and the Glover tower. This was already an open muffle concentration, which, however, depended on the existence of roasting kilns. I then tried to avoid the latter restriction by providing a separate furnace, while retaining the principle of muffle concentration for the sake of simplicity and economical use of heat."

According to the process described by this inventor in the Patent Specification under notice the concentration of sulphuric acid takes place in stages, *viz.*, a preliminary concentration, and a final or main concentration in open trays or flat vessels mounted in a closed shallow muffle, which is heated by the furnace gases at the top and bottom and on the sides. As indicated by Figs. 122, 122A and 122B the preliminary concentration takes place preferably in two rows of quartz trays or vessels *a*, which are mounted freely in the rear (upper) furnace chamber A, and communicate with each other through overflows. If great purity of the acid is not required, these trays may remain open, so that they are heated not only from below and on the sides, but also, and with them the acid contained in the trays, from the top. The aqueous vapours passing off during the preliminary concentration are conducted to the chimney together with the

gases of combustion, which besides not molesting the neighbourhood according to Lunge (*Handbuch der Soda-Industrie*, 3rd Ed. Vol. I., pages 711 and 712) has still the advantage of a favourable influence on the intensity of concentration. If great purity of the final acid is desired, the concentrating trays may be covered with lids, which have openings protected by caps, for the escape of the aqueous vapours generated. The lids consist preferably of sheet metal so thin and placed so near the surface (of the acid), that an intense effect of radiant heat is thereby ensured. The preliminary concentration may be effected also above the furnace, for instance, in lead pans, or in a concentration tower or similar appliances. The principal concentration, that is the actual high concentration, takes place in the muffle B. This consists of

FIG. 122B.

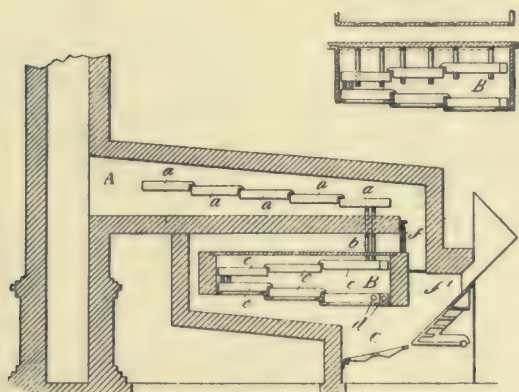


FIG. 122.

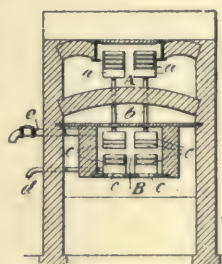


FIG. 122A.

a material adapted to resist acid at a high temperature, for instance fireproof stoneware, quartz or cast-iron. It is completely closed and heated by the fire gases from all sides as far as possible. The muffle contains open receptacles *c*, such as quartz trays, which receive the preliminarily concentrated acid through pipe *b*, concentrate it further, and then allow it to flow out through pipes *d*. The vapours of dilute acid arising during the high concentration are led off by the tube *e* to the cooling coil or the condensation apparatus. The number of quartz trays in the muffle B depends on the requirements to be fulfilled by the final concentration. The trays may be suspended, or mounted on the floor, or these two methods of supporting may be used in combination. Finally, in many cases, for instance if concentration to 97 or 98% H_2SO_4 is required, the last tray (preferably made of cast-iron)

may be sunk tightly in the floor of the muffle B, so that their lower surfaces are touched by the flame as directly as possible, without departing from the principle of concentration with muffles. The latter case is illustrated by the drawing. For more intense working the muffle may, of course, be subjected to a vacuum. The source of heat shown in the drawing is an ordinary grate C, instead of which, gas heating or semi-gas heating may, be adopted. *f* and *f* indicate sliding dampers for regulating the draught.

Zanner concludes with the observation that it has been proposed to concentrate sulphuric acid by means of a tower composed of cast-iron pipes heated externally by furnace gases, the tower containing a series of trays of earthenware or glass arranged one above the other in zig-zag fashion, and containing the acid to be concentrated, which is admitted at the top of the tower, and gradually drops from one tray to the next, being finally discharged from the lowest tray, while the vapours given off by the acid escape through a flue at the side of the tower. The trays are thus not touched by the furnace gases, but are heated indirectly by radiation from the inner surface of the cast-iron pipes. The muffle used according to the present invention is a shallow receptacle, *viz.*, substantially larger in a horizontal direction than in height, and it is heated by the furnace gases at the top and bottom, and as far as possible on the sides.

“Vitreosil” Cascade Concentrating Plant.—The success of the cascade system for the concentration of sulphuric acid may be attributed in no small measure to the introduction by the Thermal Syndicate, Ltd., of Wallsend-on-Tyne, in 1907, of “Vitreosil” vessels, by which trade name fused silica goods are known. This material is entirely proof against acids of any strength or temperature, and in this respect “Vitreosil” is unique amongst the various materials at the disposal of the chemical engineer. It is also highly refractory, and owing to its low coefficient of expansion is able to withstand sudden change of temperature without cracking. The author is indebted to the makers of “Vitreosil” for the following notes.

The first dish cascades were erected in this country and on the Continent to the plans of Benker-Hartman, and consisted of open lead dish pre-heaters, and two rows of 20 basins under closed flue covers, each row being fired independently. There were three Benker-Hartman cascade plants working in the United Kingdom in 1907-1908, when silica basins were introduced and immediately adopted in place of the porcelain vessels used prior to the advent of silica ware. In 1910 the Thermal Syndicate designed an “all silica” cascade

concentrator, having 30 shallow tray preheaters and 50 basins all under one flue, resulting in a considerable saving of fuel and ground space over the earlier plants. A reference to the illustration (Fig. 123) will show the general arrangement of a "Vitreosil" plant in which an open fire-bar furnace is employed under a portholed arch connected to the firing flue running the whole length of the plant and having a final exit to the chimney.

A scrubber is provided consisting of a lead box of about 500 cubic feet capacity packed with coke. This will give an exit down to 0.5 grain per cubic foot. Coal is usually employed as fuel, but several works are firing by Mond gas, which is of course an ideal method, since the temperature can be controlled with so much ease.

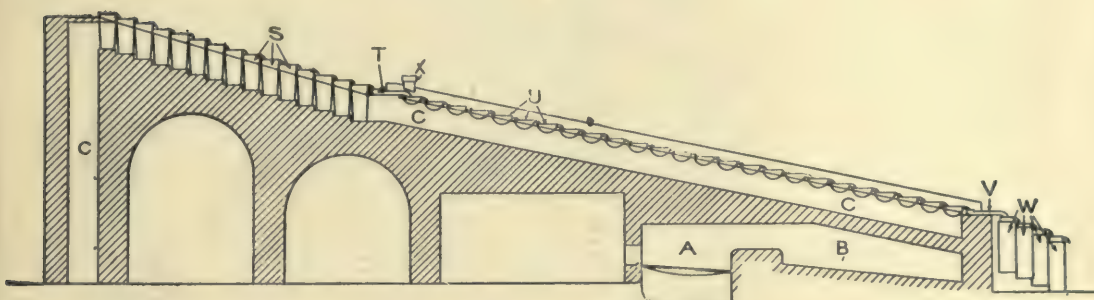


FIG. 123.—"VITREOSIL" CASCADE CONCENTRATING PLANT.

An ordinary chimney draught is sufficient, providing about $5/8$ " water suction, both for the firing and fume flues. Steam jets or fans to create draught are not required, and their use can only be necessary with insufficient draught or partially choked condensers due to the coke packing being too small grade. The latter trouble is especially common with spent acid recovery plants, where the nitro compounds deposit in the scrubber. This can be avoided by using good hard metallurgical coke, not smaller than egg size. Insufficient draught is a common cause of reduced output. For a cascade plant consisting of 30 trays and 50 basins the yield of 168° Tw. acid should not be less than $4\frac{1}{2}$ tons per 24 hours, commencing with a cold feed of 106° to 110° Tw. acid. Plants have been supplied to acid manufacturers all over the world, and to the large Government and trade explosives factories, and to those of the Allies, for the concentration of chamber acid, re-concentration of residual acids from nitration processes, and

recovery of sludge acids from mineral oil refining. At the present time (July, 1917) about 1250 units are in daily operation. There has been a good deal of controversy regarding the question of lead pans for pre-heating the weak acid, and in this connection tests were carried out on two plants operating side by side at a works in Glasgow, working under identical conditions, one being fitted with lead pans and the other silica tray pre-heaters the former with an area of 64 square feet and the latter 60 square feet. The average output of the "all silica" set was 4 tons 8 cwt. per 24 hours of 168° acid, and is often increased as circumstances require. The average output of the lead pan set was 3 tons 10 cwt. of 168° Tw. acid per 24 hours, and this quantity could not be increased without injury of the lead pre-heaters.

| | | | | |
|--------------|-------------|-------------------|---|--------------|
| Silica Trays | evaporating | 6lbs. of water | - | 6,300 B.T.U. |
| | heating | 32lbs. " " 184 | - | 5,880 " |
| | " | 94lbs. " acid 184 | - | 5,734 " |
| Lead Trays | evaporating | 5lbs. of water | - | 5,250 " |
| | heating | 27lb. " " 168 | - | 4,536 " |
| | " | 75lbs. " acid 168 | - | 4,200 " |

The first set relating to 100 lbs. of acid produced, the second set to 80lbs., this being the proportion in which the two plants were working.

The above example shows clearly the superiority of the silica preheaters over the lead, working under similar conditions. The more recent "Vitrosil" cascades are fitted with deep beaker preheaters, which latter form was adopted as possessing a greater heating surface and smaller cooling area, and giving more intimate contact with the fire gases than the shallow trays on the earlier plants. One plant operating in the Midlands, consisting of 60 beakers and 100 basins, on test during normal working gave the following results:—

Inlet Acid 106° Tw. at 90° F. No. 15 beaker 342° F. Making an increase of 242° or about 16° per beaker.

Nos. 11-15 had to be enclosed and connected to the suction flue, as the acid in this part of the preheating arrangement was in a state of violent boiling, giving off considerable quantities of acid vapour. As No. 10 beaker was at a temperature of 260° F. it may be assumed that this is about the maximum temperature allowable in the open. The output of finished acid was at the rate of ten tons per 24 hours of 94.5% H_2SO_4 .

In summing up the advantages of the silica cascade system as compared with other systems where the fire gases mix with the acid fumes, such as Kessler or Gaillard, the makers claim the following points:—

(1) The initial cost of the cascade plant is less for the same output of acid.

(2) The repairs are less and more easily executed.

(3) The acid fumes are condensed more completely and with much smaller scrubber space.

(4) The resulting acid is not liable to be contaminated with metallic impurities or flue dust.

(5) An ordinary chimney draught is sufficient without use of fans.

(6) The whole plant works more uniformly, and is not so subject to sudden variation.

The Mackenzie "Field Tube" System—An interesting development in the design of concentrating plant for sulphuric acid is the "Field tube" system of evaporating vessels patented by the late Mr. John Mackenzie, and shown in Fig. 124 (page 240). The advantages claimed for this arrangement are (1) that the vessels give ten times the heating surface of the ordinary cascade basins; (2) a plant on these lines will occupy about one-fourth the space (for a given duty); (3) one run of fifteen vessels and four ordinary basins, as shown, will more than equal a forty basin plant of the ordinary type; (4) the temperature of the flues will be very much lower, and consequently there is less chance of distilling off white fumes; (5) the very rapid circulation caused by the "Field tubes" prevents any chance of deposit, and increases very materially the rate of evaporation; (6) the cost of fuel per ton of finished acid will be less than half that of the ordinary systems; (7) the first cost of plant is less than the ordinary cascade systems.

The vessels are made in fused silica, "Tantiron," "Ironac," or lead, according to the duty required, with "Gibsonite" blocks, seatings, etc., by the Buckley Brick and Tile Co., Ltd.

The object of the tube, which is shown suspended in the long protuberance of each basin, is to promote circulation of the acid undergoing concentration. The drawing is practically self-explanatory. In a communication to the author, Mr. Mackenzie pointed out that the great fault of the ordinary cascade basin lies in the absence of any method of mingling the light acid with the heavier acid into which it falls. The change in the shape of the basin and the insertion of the simple "field tube" brings about as much intermingling as is required.

A large number of plants on the ordinary cascade system, erected to Mr. Mackenzie's designs, are at work.

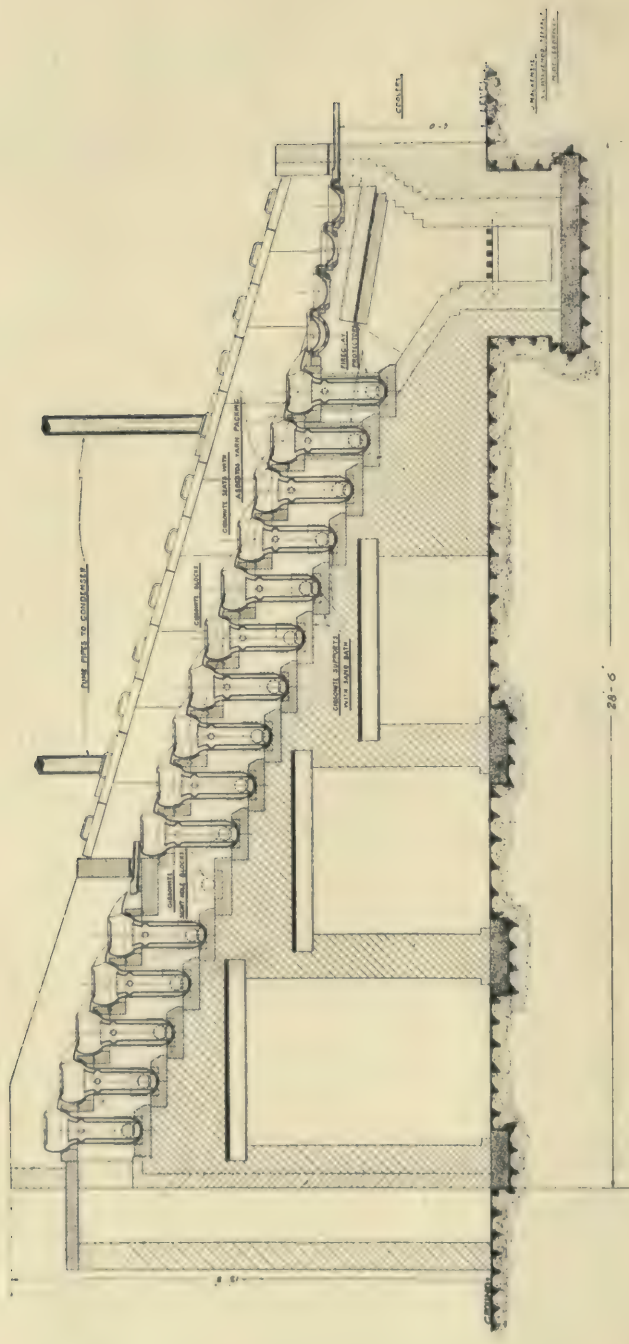


FIG. 124.—MACKENZIE "FIELD TUBE" SYSTEM CASCADE CONCENTRATING PLANT.

Falding's Chambers.—The following is the text of Falding's British Patent No. 26,452 of 1909:—

The apparatus usually employed is, besides the source of the sulphur gases, a Glover tower into which the sulphur gases mixed with air are first introduced, a series of lead chambers in which the gases are introduced after passing through the Glover tower and in which, mixed with nitrogen oxides and steam, the principal reaction takes place; and a Gay-Lussac tower from which the gases make their final exit. A series of these lead chambers are generally employed, the reaction in each being qualitatively the same but quantitatively different, the reaction in the first chamber usually being much greater than in that of any succeeding chamber and the reaction in the last chamber the least. These lead chambers are commonly made representing in their sections a longitudinal flue; their individual length being from 50 to 200 feet with a width of from 10 to 40 feet and a height varying from 6 to 40 feet. The average cross section is from 20 to 30 feet wide and from 18 to 24 feet high. The relation of the width to the height has generally been considered immaterial, being more generally dictated by problems of mechanical construction rather than by any reference to the effect of their dimensions on the working of the chambers in practical operation. In fact it has generally been recognized that a plurality of lead chambers has been necessary regardless of the individual dimensions of each lead chamber. I have found in practice by the alteration of the dimensions of the lead chamber from that commonly employed that I am able to secure an approximately complete reaction in a single lead chamber, so that I secure in a single chamber results which have previously necessitated the use of two or more chambers in series. I accomplish this result by increasing the height of the chamber relative to its length and breadth so that the height is greater than the horizontal cross section in the proportion of about 3 to 2, the height being about half as great again as the diameter or cross section of the chamber. As shown in the drawing. (Fig. 125) the gases are introduced at the top of the chamber and are withdrawn from near the bottom of the same, though I do not consider it material at what particular part of the chamber the gases are introduced, as in a chamber of the proper proportions the mixture of hot gases and steam will form a hot reaction zone at the top regardless of the point at which they are introduced into the chamber. Where I wish to increase the capacity of the plant additional chambers may be used, but these are not run in series with other chambers, but each exists as a complete unit for the production of sulphuric acid, each chamber taking its supply of gas from the Glover

tower direct so that all chambers, if more than one is employed, are run parallel and not in series. The annexed drawing is a conventional view in side elevation of a sulphuric acid plant embodying the present invention. Like characters of reference designate corresponding parts throughout the several views. The drawing represents a plant embodying my invention. A is the roaster which is represented as the source of the SO_2 employed. B represents the Glover tower through which the sulphur gases in their upward passage serve to separate from the acid running in the opposite direction through same the nitrogen oxides absorbed in the Gay-Lussac tower which will be hereinafter referred to. C represents the lead chamber. D represents a cooling tower in which the gases, after emerging from the lead chamber C are cooled preparatory to their passage into the Gay-Lussac tower E. In this tower free nitrogen oxides are absorbed by the acid running through the same, these nitrogen oxides being again liberated by the incoming sulphur gases in the Glover tower. It is to be understood that the conventional plant shown in the drawing does not represent a complete producing plant in all of its details, and to be further understood that said plant is similar in all its principles of operation with an up-to-date sulphuric acid producing plant, and that the novelty of the present invention resides in the arrangement of the lead chamber. As mentioned, the lead chamber shown at C is in the form of a tower, that is to say a chamber whose vertical axis is greater than any horizontal axis, preferably in the proportion, as above stated, of 3 to 2, although it is to be understood that the invention is not limited to any such exact proportions. While it is not broadly new to employ a lead chamber the height of which exceeds any horizontal dimension, such suggestions having been made heretofore, it was not possible to perform practically the entire reaction in one single chamber of this kind, nor have such chambers been connected heretofore singly and in parallel to the Glover tower. In construction the lead chamber is supported by any approved form of framing which is preferably of truss construction, having towers or upright truss standards jointed on their upper extremities by means of horizontal trusses which may be of substantial usual roof truss construction. The roof sheets and the side sheets are suspended, practically clear of all obstructions, and are in contact with practically no surfaces, giving free access to the atmosphere to the exterior surfaces of the lead sheets for cooling purposes. The lead lining both top sheets and side sheets contact with and are suspended only by lead suspending means.

Falding's claims are:—(1) In an apparatus for manufacturing sulphuric acid, sulphurous acid generating means, a Glover apparatus

through which the sulphurous acid gas is passed, and a single lead chamber, the vertical axis of which exceeds any horizontal axis, and a Gay-Lussac apparatus for the treatment of the gas, escaping from said lead chamber.

(2) In an apparatus for the manufacture of sulphuric acid, a Glover apparatus, a single lead chamber, having its vertical axis in the proportion of about three to two over any horizontal axis, and means for treating the products of reaction escaping from the lead chamber.

(3) In an apparatus of the character described, means for generating sulphurous acid gas, a Glover apparatus, a single lead chamber, having its height greater than any horizontal axis in the proportion of three to two, means for cooling the products of reaction, and a Gay-Lussac apparatus for treating the gas escaping from the lead chamber.

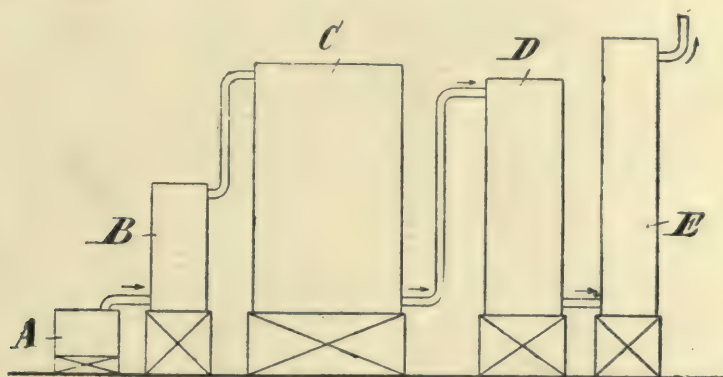


FIG. 125.—FALDING CHAMBER PLANT.

(4) In an apparatus for manufacturing sulphuric acid, a Glover apparatus and a Gay-Lussac denitrating tower apparatus, and a single lead chamber, intermediate of said two pieces of apparatus, the height of said chamber exceeding any of its horizontal axes, gas admitting means at the top of said chamber, and gas escaping means at the bottom of said chamber.

(5) In an apparatus for manufacturing sulphuric acid, a single Glover apparatus, and lead chambers whose height exceeds any of their horizontal extensions, and connected singly and in parallel to said Glover apparatus.

(6) In an apparatus for manufacturing sulphuric acid, a single Glover tower apparatus, lead chambers of greater height than any of their horizontal extensions, and connected singly and in parallel to said Glover apparatus, and gas admitting pipes at the top of said chambers, and gas escaping means at the bottom of said chambers of great height.

The Meyer Chamber.—Meyer, in his British Patent No. 18,376 of 1898, says:—"The condensation chambers usually employed in the manufacture of sulphuric acid are objectionable in that the gases or vapours to be condensed are forced to pass through said chambers on the shortest way, the speed of said gases or vapours being nearly in proportion to the cross-section of the chambers. The movement of the gases or vapours through the chambers is rectilinear and but slightly modified by the cooling action of the walls, so that said gases or vapours will enter in combination and produce acid at the passage from one chamber into the adjacent one. By this fact, the space required for the condensing chambers of the sulphurous vapours will be unduly large. It has been tried to overcome these objections by providing partitions

FIG. 126.

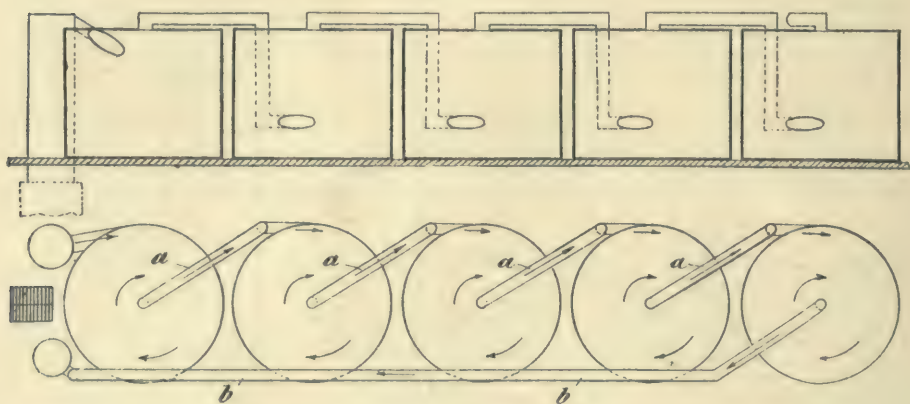


FIG. 126A.

MEYER CHAMBERS: ELEVATION AND PLAN.

or towers within the condensing chambers, which were thus divided into a plurality of smaller chambers, and in employing long conduits or similar means for causing the vapours to continue their travel to obtain a more perfect mixture of the gases and a more ready reaction. But such means have not produced the desired result, or have been very expensive, and still to-day two or four elongated condensing chambers, rectangular in cross section, are commonly used in the manufacture of sulphuric acid. This invention relates to a novel construction in such condensing chambers, the object being to force the sulphurous vapours to travel in the most convenient way, which result I attain by a peculiar arrangement of the inlet and outlet pipes of said chambers, which in this instance, are of circular or polygonal form in horizontal cross-section."

In the drawings Fig. 126 shows an elevation of a plurality of condens-

ing chambers, and Fig. 126A shows a plan view. The arrangement consists in placing the inlet pipe *a* tangentially on the side of the chambers, the outlet pipe *b* leading from the top of each chamber. "Thus the outlet pipe of one chamber will become the inlet pipe of the adjacent one, and it will be obvious that the gases or vapours may be introduced through outlet pipe *b* and expelled through inlet pipe *a* or *vice versa*. This improved arrangement results in a spiral movement of the vapours, and, when the latter are introduced through inlet pipe *a*, such movement will be gradually checked towards the centre of the chamber to be finally transformed into an upward movement. Furthermore the travel of the vapours is highly increased, whereby a greater animation among the molecules of the gases will be produced, and said animation and rotation produce a more perfect mixture of the vapours. The production of acid will be further promoted by the fact that the vapours, as they enter a condensing chamber, are thrown into frictional contact with the cooling walls of said chamber. The foregoing arrangement may be very advantageously applied to lead-chambers which are circular in cross-section. It will be obvious that the parts may be arranged in a different way, without departing from the scope of this invention."

Duron's Tower System.—The following is the text of Duron's British Patent 2408 of 1913:—"The present invention relates to a tower system for the manufacture of sulphuric acid, and its object is to prevent loss of nitric oxide as well as to reduce the consumption of nitric acid to a minimum. Tower systems are already known in which sulphur dioxide gas is passed in one direction while the towers are irrigated with nitrous sulphuric acid flowing in the opposite direction, the towers being mutually connected in pairs within which part of the acid circulates, the pairs being mutually connected through the medium of one of the towers in which the final denitrification and concentration takes place.

"The tower system according to the present invention comprises one or more concentration, denitrification, reaction and absorption towers, and its main features consist in that the reaction towers are irrigated each with its own acid, and that the nitric acid supply and most of the water supply are directed to these towers alone, part of the water being led to the denitrification tower. The acid produced in the reaction towers, and not utilised in the irrigation of these towers, is led off to the denitrification tower, which also receives the acid produced in the absorption towers, the latter being irrigated by the acid produced in the concentration tower, which again receives the acid produced in the denitrification tower,

"The irrigation method of the reaction towers, in conjunction with the features that all the nitro-sulphuric acids from the absorption tower (Gay-Lussac tower) as well as from the reaction towers, are led to denitrification towers (Glover's towers), and that the absorption towers are irrigated with the sulphuric acid coming from the concentration tower, results in the advantage that a disturbance in the working of any particular tower is confined to such tower alone, and can easily be remedied. This is due to the manner in which the towers are connected, the reaction in each particular tower being capable of regulation without influencing the others."

In the accompanying drawings the invention is illustrated by way of example Fig. 127 representing a side view, and Fig. 127A a plan of the tower system, both figures being shown partly in section. "The tower system shown in the drawings comprises six towers, which are designated *a*, *b*, *c*, *d*, *e* and *f* respectively. For smaller outputs a system of five towers will be sufficient. The hot sulphur dioxide gases, which have a high velocity, pass into the lower part of the tower *a* through a tangentially arranged inlet *g*, which causes the gases to whirl round in the tower, and distribute themselves uniformly under the grate surface *h*. The space is thus fully utilised, although the cross-sectional area of the tower is rather large compared with the gas volume. A second, also tangentially arranged, inlet *g* is only employed in cases when gas has to be led to the tower through a reserve pipe. The hot gases rise through the concentration tower *a* and concentrate the sulphuric acid, which is drawn from the tower *b*, and led downwards through the tower *a*, to at least 60° Beaumé. This degree of concentration is necessary for the subsequent absorption of nitric acid. Owing to the concentration, part of the water, as is necessary for the process, is led back through the system in the form of steam, and goes into the lower part of the second tower, the denitrification process in the latter being thereby facilitated. The gases discharged by the concentration tower *a* are driven off through three branch pipes *i* and a conduit *k* to the Glover's tower *b*, in which they pass downwards through a centrally disposed conduit *l*. At the lower end of the conduit *l* the gases pass through radially disposed outlet ports *m*, and distribute themselves uniformly in this tower under the grate *n*, which carries the filling, a complete utilisation of the space being attained also in this instance. The irrigation acid, the nitrosulphuric acids produced in the Gay-Lussac towers *e* and *f*, as well as some of the acid produced in the towers *c*, *d*, are passed off to the tower *b*, in accordance with the diagram (Fig. 127A.) In this tower *b* the acid must not exceed 57° Beaumé, since the acid should

be capable of giving off the nitric oxide at a comparatively low temperature. The sulphuric acid then flows, as already noticed, into the concentration tower *a*. If the Glover's tower *b* should fail to act satisfactorily, and if the produced sulphuric acid, therefore, should happen to contain nitrosulphuric acid combinations, the denitrification would be continued and completed by the tower *a*. The sulphur dioxide gases, which pass through the entire filling of the tower *b*, are led off at three different points of the roof through pipes *i* which open into a common conduit *k*. The gases are led off to the Gay-Lussac towers *e* and *f* in a corresponding manner. The last tower *f* discharges the gases, after the latter have been cleared of sulphur dioxide, either directly

FIG. 127.

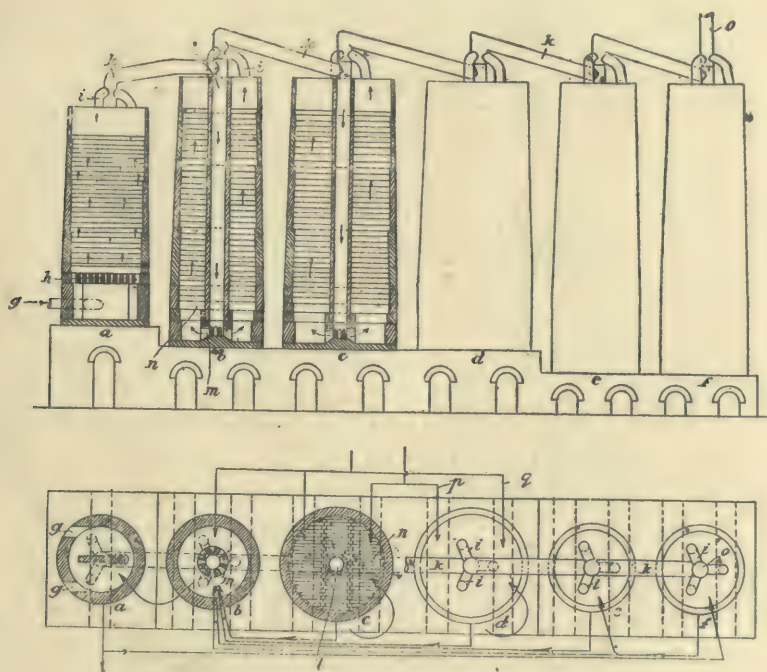


FIG. 127A.

DURON'S TOWER SYSTEM.

into the atmosphere through a discharge pipe *o*, or through a sand filter adapted to absorb the last traces of moisture.

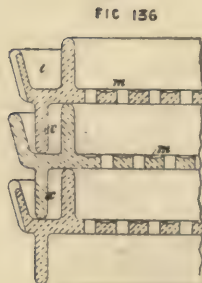
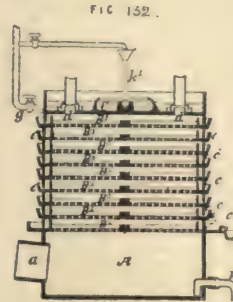
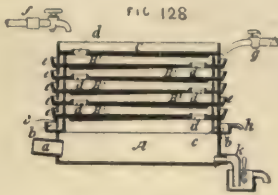
"The third tower *c* and the fourth tower *d* in which the acids are formed, are both irrigated against the gas flow, with part of the acid they themselves produce. These towers, however, are completely independent of one another, so that they can work and be regulated independently. Moreover, the nitric acid and most of the water necessary

for carrying out the process are led in suitable proportions to the towers *c* and *d* through conduits *p* and *q*, part of the water being led to the tower *b*. The acid produced in the towers *c* and *d*, in so far as it is not utilised in the irrigation of the towers, is conducted, as previously noticed, by parallel conduits to the Glover's tower *b*. The degree of concentration of the acid produced in the towers *c* and *d* must be maintained at 55° Beaumé in order to prevent the absorption or dissociation of the nitric oxide. The temperature in the interior of these towers is regulated accordingly, and the irrigation acid is, in order to reduce the reaction temperature before it is raised, cooled by suitable coolers. The absorption towers *e* and *f* are irrigated, as indicated by the diagram (Fig. 127) with part of the acid formed in the concentration tower *a*.

Duron's claims are:—(1) A tower system for the manufacture of sulphuric acid, comprising a series of towers, of which the middle ones, serving as reaction towers for the formation of acid, are adapted to receive the nitric acid and most of the water supply, and to be irrigated each by the acid produced in it. (2) In a tower system according to the preceding claim, connecting the towers so that the superfluous acid from the reaction towers, as well as the acid produced in the absorption towers, is led to the denitrification tower, the acid produced in the latter being led to the concentration tower, whose acid is partly employed for the irrigation of the absorption towers.

Lunge Plate Towers.—Figs. 128 to 136 show the Lunge plate tower as described in Lunge's British Patent No. 10,355 of 1885. In his specification, Lunge says:—"This invention has for its object improvements in apparatus for bringing gases into intimate contact with liquid or solid substances, whereby the action of the gases upon such liquids or solids is made to take place as perfectly as possible, a result required in many operations of industrial chemistry." Fig. 128 is a vertical section of the apparatus, Fig. 129 is a bottom view of part of the apparatus, Fig. 130 a vertical transverse section taken in the plane of the line α, β (Fig. 129,) and Fig. 131 a vertical transverse section taken in the plane of the line γ, δ (Fig. 129 of the same part.) "The vessel A forming the base of the apparatus, is provided with the inlet pipe *a* for the gases, and the outlet pipe *k* for the liquid, and is surrounded by a trough *b*, into which fits the the downward projecting socket *c* of the plate B¹, in order to constitute a hydraulic or water-lute or seal. This plate B¹ is likewise surrounded by a trough *e*, in which is placed the downward projecting socket *c* of a following plate B². Upon this plate B² there is placed another plate B³ of similar construction, and in the same way

any number of plates $B^1 B^2 B^3$ of similar construction may be successively superposed, one over another, always forming hydraulic lutes, between the trough e of each plate and the socket c of each adjoining plate. To these plates $B^1 B^2 B^3$ rows of holes d are arranged in such a way that these rows of holes are situated alternately on the right and on the left side of the plates. The upper ends of the holes d are surrounded



by low collars, but the remainder of the upper surfaces of the plates are plain and horizontal. The lower surfaces of the plates are preferably inclined as shown in Fig. 128 in such a manner that it slopes down from the holes d to the opposite side of the plate.

Shallow furrows or grooves *l* (Figs. 129-131) starting from the holes *d*, the purpose of which is hereinafter described and specified may be advantageously arranged upon the inclined lower surfaces.

“Supposing that it is desired to effect in the apparatus an action of gases upon a liquid. The gases in this case enter the vessel A through the pipe *a*, whilst the liquid to be treated is flowing through the pipe *f* upon the top or covering plate C of the superposed vessels. This covering plate C is analogous in construction to the other plates B¹ B² B³; but if the gases which leave the apparatus are to be conveyed to some other apparatus, to be further utilized, the holes *d* of the covering plate C are provided with hydraulic lutes connecting these holes to the pipes which convey the gases to the following apparatus. The gases ascending through the holes *d* pass the apparatus in a zig-zag line, and are thereby brought into very intimate contact with the liquid which, flowing over the low collars, which surround the holes *d*, descends from plate to plate and finally flows off in a highly concentrated state through the pipe *k*. The gases not only come into contact with the thin layer of liquid spread upon the upper surfaces of the plates B¹ B² B³, but also with the layers of liquid which adhere to the inclined lower surfaces of the plates, the liquid flowing down by adhesion from the holes *d* over such inclined lower surfaces. By this means there is a very extensive surface of contact afforded between the gases and the liquids.

“In order to form the hydraulic lutes, water or any other convenient liquid, as the case may be, is made to run through the cock *g* into the uppermost trough *e* of the apparatus. The liquid descends by means of overflows situated alternately at opposite sides of the troughs *e*, passes in this way through all the hydraulic lutes and finally leaves the trough *b*, running off at *h*. This stream of liquid through the hydraulic lutes, which may either be continuous or intermittent, serves in many cases advantageously for cooling the apparatus.

“In some cases the apparatus above described may be slightly modified in the manner represented by the Figs. 132-136 of the drawing, in which Fig. 132 is a vertical section of the modified apparatus, Fig. 133 the top view and Fig. 134 the bottom view of the covering plate C. Fig. 135 is the top view of one of the other plates, Fig. 136 is a part of the vertical section Fig. 132 on a larger scale.

“The plates B¹ B² B³ are in this modified construction of the apparatus provided with numerous small holes, the total cross-section of which is made greater than that of the gas inlet pipe *a*. The lower surface of the plates is plain and horizontal and the upper

surface may be slightly hollowed out at *m* (Fig. 136) between the small holes. The latter are arranged in alternate positions in the several plates, so that the liquid passing through the holes falls upon a solid part of the plate below and is thereby scattered about. The ascending gases are thoroughly dispersed and caused to continuously change their course by the manner of arranging the perforations of the plates, and they are intimately mixed with the dispersed or broken up and atomized liquid. In order that the liquid forming very thin layers upon the plates and being contained especially in the hollows *m*, may be spread as uniformly as possible over the plates *B*¹ *B*² and also to provide against the contingency of the plates being somewhat out of level, the upper surfaces of the latter are divided by ledges *i* into a number of entirely separate compartments. The same arrangement is also given to the covering plate *C* (Fig. 133) which may have in each compartment one or more larger holes *d* instead of a greater number of small ones. These larger holes *d*, as already stated in the description of the first apparatus represented in Fig. 132, can be easily connected by means of water lutes with the pipes which convey the gases to some other piece of apparatus. The liquid may advantageously be spread over the surface of the covering plate *C* by means of a reaction wheel *k*¹ (Fig. 132) made to revolve by the liquid issuing from it; and in order to distribute the liquid uniformly over the plate below, furrows *n* (Fig. 134) may be made, extending radially from the holes *d* on the lower surface of the covering plate *C*.

"If in the improved apparatus it is required that the gases should act upon solid bodies or mixtures of solid and liquid bodies, the plates *B*¹ *B*² may be singly filled with the solid or the mixture of solid and liquid substances, and superposed one over another and as soon as the operation is ended, they may be lifted off and emptied, one after the other.

"The apparatus shewn in Fig. 128-131 is more appropriate for treating solid substances in the form of powder or mud, whilst the apparatus shewn in Figs. 132-136 is more convenient for treating coarsely granulated substances. The apparatus may be constructed of stoneware, earthenware, metal, wood, or any other suitable material.

"Instead of the square or circular shape of the plates *B*¹ *B*² shown by the drawings any other form may be employed.

The claims are :—(1) The combination, in an apparatus for effecting the action of gases upon liquids or solids, of the vessel *A*, the superposed plates *B*¹ *B*², etc., and the covering plate *C*, all connected, and if required also cooled, by hydraulic lutes, substantially as set forth and shown. (2) In the superposed plates *B*¹ *B*² *B*³, etc., (Figs. 128-131) the

inclined lower surfaces substantially as described and for the purpose specified. (3) The superposed plates B (Figs. 132-136) provided with numerous small holes arranged in alternate positions in the successive plates, substantially as and for the purpose specified. (4) The arrangement of ledges *i* upon the surfaces of the superposed plates B¹ B², etc., and the covering plate C, substantially as described and for the purpose set forth."

Lunge and Rohrmann's Specification of British Patent No. 10,037 of 1886 describes improvements as follows:—

"The present improvements consist first in the particular method of arrangement of a number of superposed plates B¹ B², etc., the apparatus being in two parts, that is to say the syphon or traplike trough P (Fig. 142), which receives loose the perforated discs or plates B¹ B² B³.

"An improvement in the said troughs P consists in the application of a downward projecting socket H¹ K¹ which prevents drops of liquid from entering into the trap P, any such drops flowing along the under side of the plates B to their outer end and then falling down from the socket K¹. The level of the water in these troughs will remain therefore constantly the same. Hitherto such drops formed and arriving at the outer end of the plates B by their cohesion to the flat under side, filled by degrees the troughs with the absorption liquid. No liquid will become lost in this way by this new method of construction.

"Another improvement relates to the construction of the perforated plates B. Fig. 137 represents a partial upper view of such a plate B. Fig. 138 is a vertical section of Fig. 137. Fig. 139 is a view from beneath. Fig. 140 is a vertical section of a modification of Fig. 138, and Fig. 141 is a second modification of Fig. 138.

"Fig. 142 represents a vertical section of a so-called absorption turret formed by the vessel A at the base of the apparatus with its hollow ring or trough in which the downward projecting socket of the superposed vessels P forms a trap or hydraulic lute. The upper end of this turret is closed by the helmet or cover *f* and the cap R with its inside sieve S. J are the troughs into which the superposed vessels dip. Instead of dividing (as in the former Patent already alluded to) the surface of the plates B into sections the upper face of such plates is divided into squares by small lintels or ribs F (Fig. 137) of a convenient height, of the same material as the plates. The inside of each square has in its centre a small round or oval perforation G (Fig. 137) which is provided on its margin with a small rim H, not quite so high as the aforesaid ribs F. In this way the liquid will be forced to remain in the

basin so formed, until it overflows, by passing over the rims H and through the holes.

"The three aforesaid systems of plates B may be employed either together, or one of these systems alone. The liquid is forced to flow off from each plate to the one below in a perfectly regular and uniform manner. In order that the liquid may not cohere to the under surface of each plate and run together into large drops, the lower face of these plates is formed in the following manner. Grooves are formed (see Fig. 139) corresponding with the position of

Fig. 138.



Fig. 141.



Fig. 140.



Fig. 137.

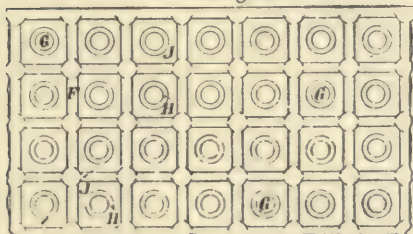
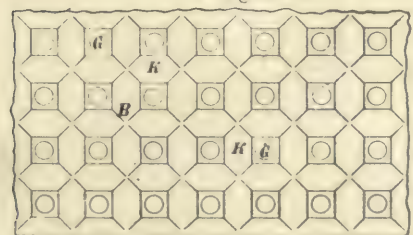
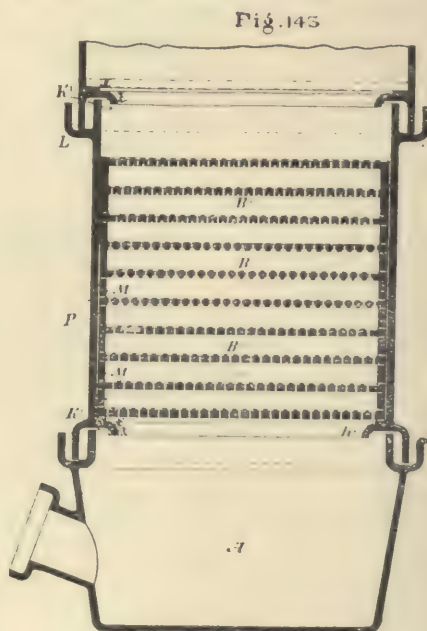
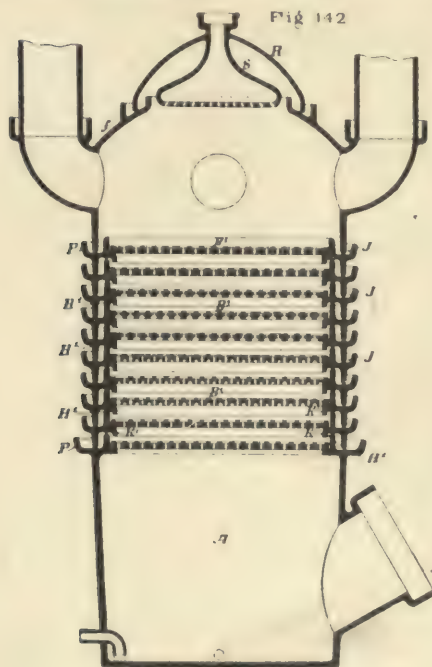


Fig. 139.



the ranges of holes, in both directions, that is to say, these channels or grooves cross each other. By this means the remaining part of the material forms in the entire thickness of the plate's downward cone shaped projections on the ends of which the liquid overflowing through the holes G accumulates, forming drops, which finally fall down to the plate below.

"In the modification represented by Fig. 140 the holes G are placed in the cones described, formed by the channels or grooves, by means of which construction the liquid will be forced to fall drop by drop down to the following or next lower plate the holes of which are arranged in such a way that they do not correspond with those of the next lower one and so on. The squares already described obtained and formed by the ribs F (Fig. 137) may be made of any desired sizes, or there may be made two, four, six, or other number of holes in each such square. By means of this arrangement the contact and action of gases upon liquids or solids takes place in the most effective and perfect manner.



"In Fig. 143 is shown a modification of an entire element or small turret. The vessel A remains the same as already described and also the method of forming the trap or hydraulic lute, the socket K' consists in this arrangement of an enlarged shoulder X and a nose Z, receiving the first plate, upon this plate is placed a ring M upon which rests the second perforated plate and so on, until the top of the cylinder, viz., the helmet R.S. (Fig. 142) is reached. L is a trough to receive the cover. The supply of liquid to the turrets may be effected by a helmet especially constructed with a perforated plate or rose. The perforations in this rose are about half a millimetre in diameter and are divergently directed, so that the whole surface of the uppermost plate is evenly and

symmetrically supplied, and consequently all the following plates are also similarly symmetrically supplied, this being a condition of great importance for the effective working of the turret."

The claims are :—(1) At the syphon the arrangement of the inner downward projecting sockets K^1 to prevent the descending liquid from entering into the syphon, substantially as described and shown. (2) The division of the plates B^1 , described in the former Patent already referred to into two portions, that is to say into the annular syphons J, and the perforated plates B^1 B^2 B^3 , as before described. (3) The separation of every perforation on the upper surface by small ledges or ribs, forming a basin around the hole and at the lower surface the arrangement of small channels around every hole, substantially as described and shown. (4) The arrangement of the rims H on the plates surrounding the upper surface of the plates, to produce a constant level of the liquid contained in the small basins, all substantially as herein described and shown. (5) In the helmet R the arrangement of a rose S fitted with divergent perforations in order to effect a symmetrical feeding of liquid upon the upper plate, and consequently the symmetrical feeding of all the following plates, all substantially as described and shown.

Fig. 144 shows the modification described in Lunge's British Patent 6989 of 1889. Referring to Fig. 144, 1 is a central vertical section of a "plate-column," 2, 3 and 4 are plan views of three modifications, respectively, of the plate supporting rings or brackets; 5 is a cross section of a plate supporting ring; 6 and 7 are a plan and side elevation respectively, of one modification of the standing joint of the segments composing the supporting ring; 8 and 9 are a plan and side elevation, respectively, of another modification of the segment-joint; 10 and 11 are a plan and side elevation respectively of a third modification of the joint; 12 and 13 are plans of the upper and lower surface respectively of a perforated plate; 14 is a cross section, showing three modifications of the perforated plate, and 15 and 16 are a cross section and plan, respectively, of another modification of perforated plate.

In the previous patents already referred to, the jacket surrounding the "plate column" was described to consist of the same material as the perforated plates proper, but according to the present improvement the jacket *a* is constructed of a suitable metal, such as lead, or of slabs of suitable stone, or lava, or a similar material, while the plates *b*, are made, as before, of burned clay, such as stoneware or the like, the perforations of the plates being preferably arranged in such a manner as to compel the liquid, while dripping down, to take a determined course from one plate to the next one, the plates carrying webs which facilitate

the escape of the liquid from the surface of each plate down to the succeeding one, without remaining on the plates for any length of time.

Where columns of cylindrical shape are being used, the perforated plates *b* are each supported and kept apart by annular supporting brackets *c*; such brackets or rings may consist, each, of one solid piece, or they may preferably be composed of several segments, as shown in 2, 3, 4 and 5, Fig. 144; in the latter case they need not occupy the entire circum-

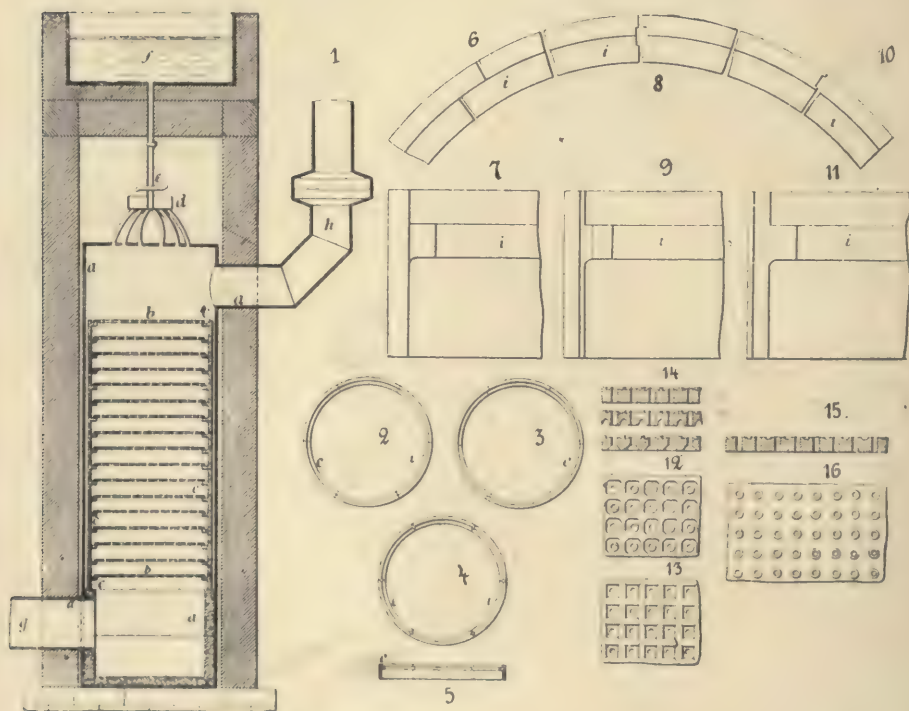


FIG. 144.

-ference of the column, but support the plates only at several parts of their rim. With square, rectangular, or otherwise shaped columns, the brackets are made corresponding to the cross section of the column.

"In the drawings the reservoir *f*, containing the absorbing liquid, is shown arranged at the top of the column; from the said reservoir or tank the liquid passes down through a pipe *e* to a reaction wheel *d*. The gas to be treated enters through the pipe *g* and escapes through

the pipe *h*. All these parts are only indicated for the purpose of better understanding the invention, and they form no part of the same nor of the principle of the apparatus; the feed of the liquid, as well as the admission and exhaust of the gas may be varied according to the requirements of practice.

"The purpose of the improved construction, as before described, is the following:—In many cases a jacket made of metal or stone is preferable to one made of earthenware (stone-ware) or the like, partly for the reason that it offers greater convenience and facilities in constructing the jacket (for instance with square or rectangular columns), and partly because it is not so liable to become creviced, but may effectually be kept tight, so as to prevent any escape of gas. Of course, the material of which the jacket *a* is made must be of such a kind as to resist any influence of the chemical agents which may enter into contact with the said jacket. In the manufacturing of sulphuric acid, a jacket of lead will answer very well, while in the manufacturing of nitric or hydrochloric acid the lead jacket would not do. On the other hand, it has often been experienced as a defect that the inside lining of a column is more subject to wear and tear by chemical action than its jacket. This is for instance the case with leaden plates, for the reason that such plates are not protected, like the jacket, by the cooling action of the air from outside, or even with coke, which may work an influence on some gases. In all such and similar cases the improvements of the present invention may be usefully employed, because they provide an inside lining to the jacket, which resists the chemical influence of the gases and at the same time provide a jacket not liable to become leaky by crevices, which jacket, moreover, may be protected against all immediate contact of the gases by means of the supporting brackets *c*. The apparatus as described is particularly adapted for use in manufacturing sulphuric acid, serving as a Gay-Lussac column or Glover column, or, especially, by being arranged between any two lead chambers of the system, in exchange for the connecting pipes usually employed. By the use of the improved apparatus, a more intimate mixture of the gases is obtained, as also the condensation of the vapours of liquid floating about; in consequence a superior reaction of the liquid matter on the gases is obtained, all of which tends to effect a considerable saving of chamber space.

"The annular brackets or plate supports *c* may consist, each, of several segments; in 2, 3, 4, and 5, six segments are shown, the joints of which are either in a line 2, or overlapping each other 3, 6, and 7, or with a groove between them 4, 8, 9, 10, and 11, which groove may be filled up by molten lead or any other suitable substance for the purpose of better preventing the escape of gas through the joints. Such

additional tightening substance, however, will scarcely be necessary in general use. The annular brackets *c* have a web or rib *i* formed to them and projecting inside, and on the said web the perforated plate *b* loosely rests and there is no pressure on the plates, the brackets *c* supporting each other by themselves. By this means the column may be constructed up to any desired height, without risk of the lower plates being cracked by the pressure of the upper column. The rings or brackets *c* may have a suitable packing, lute, or cement, applied between them to prevent the gases from escaping and entering into contact with the jacket"

Petersen's "Ring" System.—Petersen's "ring" system of towers and chambers is described in his British Patent No. 27,738, of 1907. In his specification Petersen states that his invention "has reference to means for increasing the efficiency of the reaction spaces in the lead chamber process of making sulphuric acid by providing for a vastly increased amount of oxides of nitrogen, circulated in the system, without overtaxing the capacity of the system. It is a well known fact that the acid producing efficiency of any given system of lead chambers increases chiefly in proportion to the amount of oxides of nitrogen which are kept in circulation in the system. This principle has already been utilised by increasing the volume of the Gay-Lussac space of the lead chamber system considerably as compared with the previous method of working, which resulted in a corresponding increase of the Glover space of the particular system for the purpose of complete denitration of the nitrous vitriol obtained in the Gay-Lussac space. There is however a limit to the increase of the capacity of the Gay-Lussac space—these limits varying with the conditions of the Glover space. The size of the Glover tower or towers is limited, inasmuch as the dimensions of the said towers must be kept down and within such limits as to enable the Glover apparatus to effect the necessary concentration and practically complete denitration, so as to yield an acid which is fit to go on the Gay-Lussac towers. This limit will be reached when the acid obtained from the Glover apparatus ceases to be perfectly denitrated and has gone down below sixty degrees Beaumé. With a given quantity of sulphurous acid gases only a certain definite amount of nitrous vitriol can be treated under the conditions prevailing in the system. Whenever this quantity is exceeded, the acid will pass off from the Glover apparatus with a certain percentage of nitrous vitriol, provided the acid has maintained its strength, or, in case perfect denitration of the nitrous vitriol has been attempted, the strength of the sulphuric acid obtained will have gone down too much,

on account of too much steam or dilution being used in the denitrating process, so as to render such acid incapable of absorbing the remaining traces of nitrous acid on the Gay-Lussac tower or towers.

"In view of this state of the art, the conclusion would appear to be justified that there is a certain limit to the amount of oxides of nitrogen to be kept in circulation in a given system, unless special means are provided for bringing up the weak Glover acid obtained in the case hereinbefore mentioned, where complete denitration was aimed at, to its correct concentration by a separate concentrating process. Aside from the considerable costs of plant incurred thereby, this way of procedure would entail quite considerable costs of operation. Assuming for instance a daily circulation of about one hundred thousand pounds of Glover acid, this would entail a cost of, say, five pounds a day approximately, the costs of concentration for one hundred pounds being taken on an average to amount to about a little more than one penny.

"There is, however, a possibility of augmenting the efficiency of any given lead chamber system, consisting of any given number of reaction spaces, to any desired extent, by increasing the amount of oxides of nitrogen introduced into the system, and without the inconveniences above referred to, and without any necessity of evaporating a large amount of dilute Glover acid, this purpose being accomplished in this invention by combining the usual system of Glover spaces and Gay-Lussac spaces rinsed with the ordinary strong nitrous vitriol of sixty degrees Beaumé strength and ordinary strong vitriol respectively, with another system of Glover spaces and Gay-Lussac spaces rinsed with weaker nitrous vitriol and weaker vitriol respectively, circulating on this second system only, and which may be obtained from any convenient source, the strength of the acid of the said second system being such as to enable such acid to absorb readily the lower oxides of nitrogen on the one hand, and of liberating the same again on the other hand, by suitable treatment, without the necessity of employing unduly high temperatures and without any undue or excessive dilution. This property, which is not shown by the ordinary approximately 60° Beaumé acid, inasmuch as this acid in the state of nitrous vitriol can only be denitrated by the application of a high temperature and by dilution, is peculiar to a weaker acid the strength of which ranges say, from 54 to 55° Beaumé approximately. This weaker acid is made use of for the rinsing of the second system of Glover and of Gay-Lussac spaces. The more or less large excess of oxides of nitrogen, escaping from the reaction chambers in the case of an increase of the nitric acid or nitre fed to the system, is absorbed

almost entirely by the Gay-Lussac spaces which are rinsed by the weaker acid. When the weaker nitrous vitriol obtained thereby, instead of being fed to the Glover tower of the other system which is fed with ordinary 60° Beaumé nitrous vitriol acid obtained from the Gay-Lussac spaces of the said first system, is submitted to a separate denitrating process, which latter denitrating process, on account of the dilution of said nitrous vitriol acid, takes place very rapidly and easily, the vapours of oxides of nitrogen which escape at the end of the system will be recovered and will be utilized over again in the sulphuric acid making process, and, on the other hand, any dilution of the strong Glover acid, fed to the Gay-Lussac spaces of the first system on which the complete absorption of the oxides of nitrogen is to be effected is also avoided thereby. Thus, by this invention means are provided of facilitating the admission and increasing the amount of oxides of nitrogen to be fed to the lead chamber system, and of recovering the entire amount of oxides of nitrogen that might have escaped the reaction in the various reaction spaces, and to prevent the escape of such oxides, without however interfering in any way with the concentration and the ready denitration of the nitrous vitriol obtained in the Gay-Lussac spaces of the first system and of the Glover acid resulting therefrom. Any oxides of nitrogen which might have escaped absorption by the weak acid of the Gay-Lussac spaces of the additional system which is so arranged as to succeed directly to the reaction spaces, are readily absorbed by the then succeeding Gay-Lussac spaces of the first or original system, and the acids which might have become diluted by water vapours, escaping from the lead chambers, are brought up again to the required strength by the formation of sulphuric acid occurring in the denitrating Glover tower or towers. Hence, there will not be any appreciable dilution of the acids of the second weakly rinsed system. It will therefore be possible to maintain the concentration of the acids of different strengths, circulating on both systems, in a perfectly automatic manner.

"Among the various ways of arranging the different pieces of apparatus of a sulphuric acid making plant according to my invention, I may mention the following arrangement of succession of towers and reaction spaces:—(1) Glover tower, rinsed with 60° Beaumé nitrous vitriol and with dilute acid; (2) denitrating tower, rinsed for instance with nitrous vitriol of from 54° to 55° Beaumé, coming from the Gay-Lussac towers of the second system; (3) lead chambers or other similarly acting reaction spaces; (4) Gay-Lussac tower or towers of the second system, to be rinsed with Glover acid of from 54° to 55° Beaumé; (5) Gay-Lussac tower or towers of the first system, to be

rinsed with a 60° Beaumé Glover acid. In this system, herein mentioned as an instance of carrying the invention into practice, the Glover and Gay-Lussac spaces (1) and (5) constitute the first system, forming, so to speak, a kind of exterior ring; the Glover and Gay-Lussac-spaces (2) and (4) form the second system, constituting, so to speak, an interior ring, and serve for the absorption of the excess of oxides of nitrogen or of part of the said oxides, escaping from the reaction chambers (3) at the end of the same, and for liberating said oxides again, so as to be used over again in the said reaction chambers.

"I am aware that it has been suggested heretofore to arrange upright cases or columns or the like between the Glover and the first chamber, and between the last chamber and the Gay-Lussac towers, such cases or columns containing cooling pipes or the like, and being rinsed with dilute sulphuric acid, and causing the reagents to become intimately mixed and the small vesicles of acid already formed and kept suspended in the atmosphere of the chambers to become condensed and collected, on account of the cooling and on account of the obstruction placed in the way of said reagents and the like by said cooling pipes or the like; but this kind of arrangement is entirely different from my invention and cannot be used for the purpose of the latter, inasmuch as in my invention it is necessary that the gases and other reagents contained in the atmosphere of the sulphuric acid plant pass through a comparatively high tower, and then it is necessary to rinse the reaction space which is interposed between the chambers and the Glover spaces not with dilute sulphuric acid, as in the arrangement referred to, but with nitrous vitriol which is obtained in the reaction space interposed between the last chamber and the usual Gay-Lussac space, and furthermore than that while the acid used in my invention for the operation of the additional system of spaces is of less strength than the acids used in the ordinary first system, it is not properly speaking a dilute acid; nor is it necessary to cool in the space interposed between the Glover spaces and the chambers; heating being necessary on the contrary for effecting denitration in the space interposed between the Glover spaces and the chambers."

The claims of Petersen's Specification are:—(1) The herein described method of increasing the intensity and efficiency of the reaction spaces in the lead chamber process of making sulphuric acid, and characterised by the fact that the Glover-spaces and the Gay-Lussac-spaces of the plant are divided respectively into two sections, sets or series, one of said sets or series being rinsed with ordinary strong nitrous vitriol, and with strong Glover acid respectively, while the other sets or series are rinsed with weaker nitrous vitriol or acid respectively of such

strength as to be enabled to absorb oxides of nitrogen on the one hand, and on the other hand to liberate the same again at a comparatively low temperature.

(2) Sulphuric acid manufacturing plant for carrying into practice the method, as set forth in the first claim, and characterised by two different pieces of Glover apparatus, separate from each other, succeeding the sulphurous acid generating means, and preceding the chambers, and two different pieces of Gay-Lussac apparatus, separate from each other, and succeeding the chambers, and separate liquid delivering and admitting means between each set of Glover apparatus and each set of Gay-Lussac apparatus.

(3) Method and arrangement of apparatus, as hereinbefore set forth and described.

The Opl Tower.—In the specification of their British Patent No. 20,171 of 1908 (Erste Oesterr. Sodafabrik and Carl Opl) the patentees observe that the old chamber process for the manufacture of sulphuric acid is being more modified for increasing the rate of production by hastening the oxidation of the sulphur dioxide of the pyrites burner gases by bringing them into intimate contact with nitrous sulphuric acid. The sulphur dioxide from the burner gases is completely oxidised by nitrous sulphuric acid at ordinary temperature if the gases are passed through a series of vessels containing nitrous sulphuric acid. In the first vessel the sulphur dioxide is oxidised to sulphuric acid, and the nitric oxide thus liberated is oxidised to higher oxides of nitrogen by the excess of oxygen contained in the burner gases, which oxides are again taken up in the last absorption vessel. By changing the order of the vessels or the direction of the gas current, nitrous sulphuric acid is caused to flow in the contrary direction to the current of gas. Instead of absorption vessels, absorption towers can be used down which the nitrous sulphuric acid flows from a sprinkler in the opposite direction to the flow of the gases. This method is already known, but has not been applied because the towers used did not afford sufficient contact surface between the nitrous sulphuric acid and the gas. If however, according to the present invention for working the emulseur that lifts the nitrous sulphuric acid to the top of the tower there be used compressed burner gases instead of compressed air, an intimate mixture of gas and acid is made, and by atomising the nitrous sulphuric acid on the tower a complete distribution of the acid over the whole contents of the tower is obtained, so that the burner gases flowing through the tower are quickly oxidised and the nitrogen oxides re-oxidised

and absorbed. In the first tower the nitrous sulphuric acid is completely denitrified, an acid of 60° Beaumé specific gravity being formed, a part of which is used for other purposes, while part is used to absorb the oxides of nitrogen in the last tower. The water necessary to produce acid of 60° Beaumé specific gravity is sprayed in at parts of the apparatus which are not washed by the acid, so as to prevent stoppages owing to the formation and deposition of chamber crystals. Since the complete oxidation of the burner gases occurs in proportion to the rapidity with which the nitrous sulphuric acid is circulated, it is advisable to atomise an abundant supply of nitrous sulphuric acid into the tower. For this purpose the mode of conducting the process described above can be varied by using both compressed burner gases and compressed air, instead of the former alone, to work the emulseur for raising the acid and

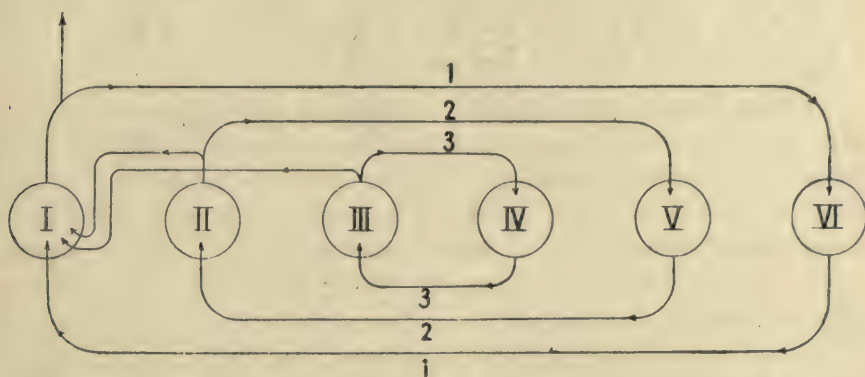


FIG. 145.

to atomise it. The burner gases used for the emulseur are also oxidised by mixing with the nitrous sulphuric acid and the production of sulphuric acid is thus increased. The emulseur may also convey to the tower the water necessary for the process as well as the nitric acid. Moreover the acid mixture contained in the emulseur may be cooled by air or water flowing over the latter. The practical working of this process, however, is only possible if the reaction towers employed are connected together in a certain manner. Experiments in this point have shown that specially advantageous results are obtained by using six towers which are connected together in the manner illustrated in the accompanying diagram (Fig. 145) in which the Roman numerals represent the six towers, and the connecting lines numbered 1, 2 and 3, the three circulations. The first tower, into which the hot burner gases

are delivered, receives the nitrous sulphuric acid from the sixth tower, and the acid produced in the other towers denitrifies it and gives up a part of the sulphuric acid produced in it to the sixth tower; the second tower receives the nitrous sulphuric acid from the fifth tower, partially denitrifies it, and gives up a part of the partially denitrified sulphuric acid produced in it to the fifth tower and a part to the first tower, and in a similar manner the third and fourth towers operate together. In this manner a three-fold circulation of the sulphuric acid is formed. In order to render uninjurious or to utilise the heat developed in the process, there may be inserted in front of the system a concentration tower for producing a sulphuric acid of 60° Beaumé specific gravity. By following the mode of operation described the patentees state that "the production of sulphuric acid in reaction towers sprayed with nitrous sulphuric acid, which production is in itself known, can be carried on commercially and the intensity of the process is so increased that the yield of a given reaction space is about ten times as great as that of a corresponding space in lead chambers."

According to E. Hartmann (*Zeitschrift für Angewandte Chemie*, 1911), the Opl Tower System had then been in operation in the works of the Ersten Österreichischen Sodafabrik at Hruschan for the past four years, and in describing the plant he points out that it is composed of two sets of towers, (1) in which the acid is formed, these acting like Glover towers, and (2) in which the oxides of nitrogen are removed, these acting as Gay-Lussac towers. The number of towers working like Glover towers is the same as those working like Gay-Lussacs. Thus, in a system for the production of 18,000 kilos. of acid of 60° Bé., six towers are used, three as Glovers, and three as Gay-Lussacs. These may be arranged horizontally or may be superimposed, according to local conditions. With the climate prevailing in Mid-Europe only the first three need be enclosed, the remainder being in the open air. In order to have as great an efficiency as possible per cubic metre, special arrangements are made to divide up the gases as completely as possible as they pass up the towers, and the strength of the construction of the towers is about the same as that ordinarily employed for Glover and Gay-Lussacs. The introduction of the acid into the towers is directly effected by elevators without the use of any high tanks. As to the working of the system, in brief this consists (as described in Opl's patent specification already quoted) in passing the acid from the first, second, and third towers to the fourth, fifth and sixth, and in the contrary direction, the first and sixth, the second and fifth, and the third and fourth communicating. Tanks at the bottom of the towers, such as are used in the

chamber system, are not required, since the acid flowing from any particular tower goes directly into the elevators. The elevator piping going to the third, fourth, fifth, and sixth towers is cooled by water. The acid produced is collected in a large tank, and finally passed through the first tower again, which leaves it concentrated to 60° Bé., and free from traces of nitrogen compounds. Above the fifth and sixth towers is a coke chest, and through it the gases pass, in order to remove all "acid fog" from them, a content of 0.5 gramme of sulphuric trioxide per cubic metre being then present on the average. In order to effect the necessary circulation, a fan is placed at the end of the plant. For a system to produce 18,000 kilos. of 60° Bé. acid per day, a total volume of 6,000 cubic metres are necessary, costing in Germany (in 1911) 100,000 to 110,000 M., coke being used as filling material, and not Glover rings, or the like. Hence 1 cubic metre produces 30 kilos. of 60° Bé. acid per 24 hours. Ground space 320 square metres. The acid produced is 20% in the first, 30% in the second, and 50% in the third tower. For acid of 36° Bé., the nitre consumption is 0.75%, calculated on chamber strength acid, and the power required is about 3 kilowatts per hour. As to the advantages over the chamber system, these are claimed to be (1) smaller cost of plant; (2) less ground space required; (3) easier supervision; (4) less skilled attention necessary; (5) production of all the acid in 60° Bé. strength.

The Suspended Lead-Work System of Vitriol Chamber Construction.—Figs. 146 to 149 show the suspended lead-work system of chamber construction as carried out by Messrs. Simon Carves, Limited, of Manchester. In this system of construction the old method of timber framework for the chambers is discarded altogether, and it is claimed to be possible to work on a chamber space of 6 to 8 cubic feet per pound of sulphur burned in the kilns, as against 14 to 20 cubic feet in the old type of chamber plant. It is further claimed that by the adoption of the suspended lead-work system the wear and tear on the lead work is reduced by about 75%, and in addition considerable economy of ground space is effected.

As will be seen in Fig. 146, the first point of departure from the old style of construction consists in housing the chambers. As will be clear from the illustration, the plant is completely enclosed, the building consisting of a skeleton steel framework, filled in with 4½-inch brickwork, pigeon-holed for the free admission of air throughout the plant. The plant illustrated has a capacity of 800 tons of pyrites acid per month, the photograph showing the burner house gable, the

highest portion of the building housing the Glover and Gay-Lussac towers, and the chambers occupying the remaining portion on the left. The plant as shown was designed for the use of liquid nitric acid in the Glover tower, a carboy hoist being provided in the tower house. The lighting of the buildings is by central glass louvres, and also from the pigeon-holing in the side walls, light being reflected from the lead sides.

The lead work is fixed on the "Moritz" system, the sheets comprising the sides and ends of the chamber being suspended freely by steel rods or gills, covered with lead and burned to the side and end



FIG. 146.

sheets, the upper ends of the steel suspension bars being slung from the steel principals. By this method of suspension the lead sheets are not rigidly fixed at any point, and expansion and contraction is entirely free from restraint. The radiation of the heat of reaction is assisted by the gills on the sides of the chamber and by the open surface of lead, which is swept by the air currents from the pigeon-holing in the walls. The chamber tops are made segmental in section, also supported by gills, giving increased strength and forming a suitable surface for the convection currents of gas in the chambers. The

chamber dishes are made with lead-lined steel sides, and the bottoms are formed by the chamber platform. A large radius is given to the junction of the sides and bottom, and an air passage is left in the floor for cooling the acid in the dish. Water atomizers of glass or platinum-iridium are fitted in the tops of the chambers, which are made

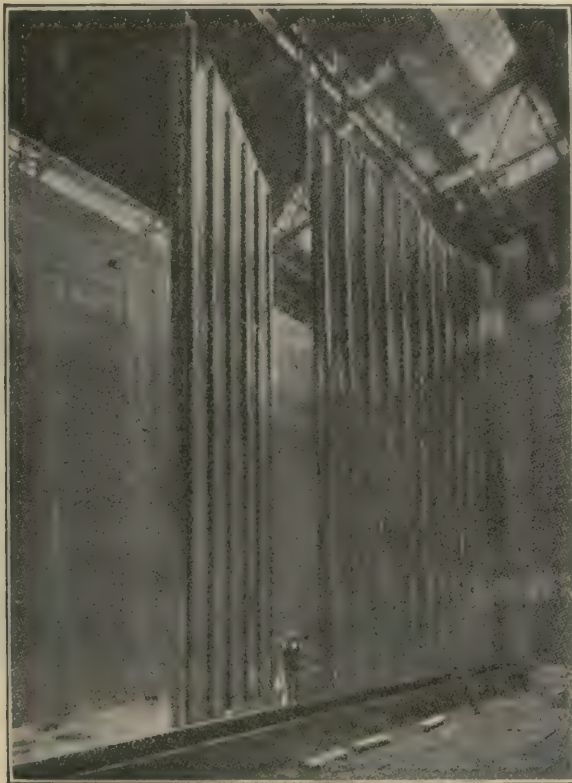


FIG. 147.

about 18 feet wide by 43 feet high. Fig. 147 shows a chamber during construction, Fig. 148 the chamber tops, and Fig. 149 shows the distribution floor above the tower.

The Glover and Gay-Lussac tower lead work is suspended from the joists carrying the distribution floor of the tower house.

The towers are usually of circular section, lined with heat and acid resisting material, and standing on a steel plate platform supported by joists resting on brick walls. Two Gay-Lussac towers are employed, with a tail fan placed in the connection between the towers.

The plant constructed as described is practically fireproof, the only timber used being in the floor boards in the bottoms of the chamber dishes and the distribution floor. Apart from the advantage of practically fireproof construction, by the elimination of rigid timber

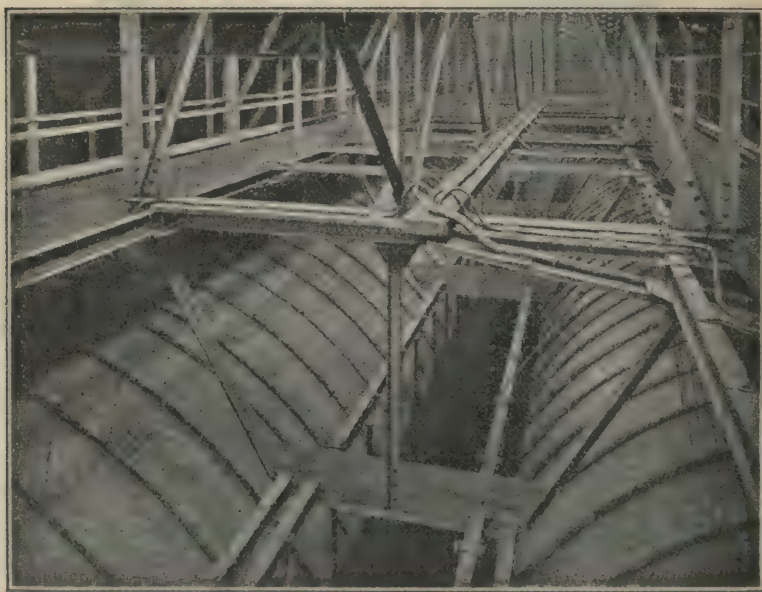


FIG. 148.

framework the nailing of the lead sheets is also avoided, and a source of weakness by reason of the continual strain on the lead work involved by the nailing of this at numerous points, with resultant fatigue and crystallisation of the lead, is also avoided. The same remarks apply to the parts of the plant exposed to the weather, such as the chamber tops. Moreover, in the old timber framework construction, radiation from the lead sheets is prevented by the presence of the timber framework, and the suspended lead system also renders the carrying out of repairs a much easier matter.

It is pointed out by Messrs. Simon Carves that efficient cooling of the lead, solidity of support, with freedom for play, are essential conditions for securing the long life of the leadwork of vitriol chambers. The presence of vibration is particularly detrimental, and all these essential conditions can be secured by the adoption of the suspended lead-work system.

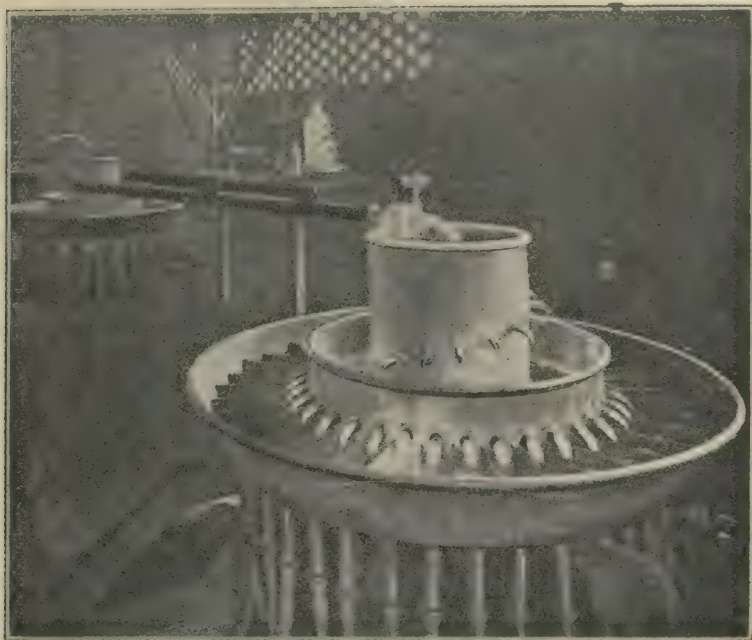


FIG. 149.

The Carmichael and Guillaume Tower Plant.—Figs. 150 and 151 show the plant of Messrs. Carmichael and Guillaume for the production of vitriol by the tower system. In a communication to the author Messrs. Carmichael and Guillaume point out that the manufacture of sulphuric acid by the tower system, totally excluding lead chambers, consists in subjecting sulphur dioxide produced by the roasting of pyrites or other sulphur-bearing material to an absorption reaction produced by intimate surface contact with sulphuric acid in process containing nitrous and nitric acids.

The reaction of the sulphur dioxide on the oxidizing acid takes place in accordance with the well-known equations :—

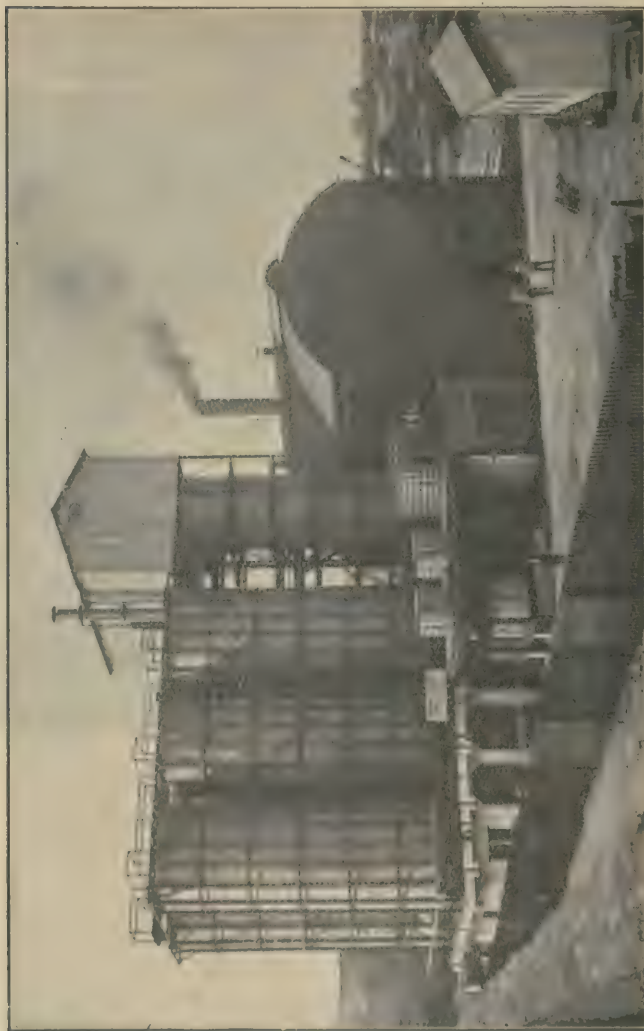
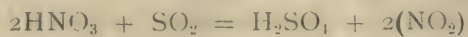


FIG. 150.

by which the sulphur dioxide is oxidized to sulphuric acid and augments the acid in process, whilst the nitrous acid is reduced to nitric oxide in the form of nitroso sulphuric acid. In turn this nitric oxide is recon-

verted into nitric and nitrous acid in the presence of oxygen and moisture and sulphuric acid, thus,

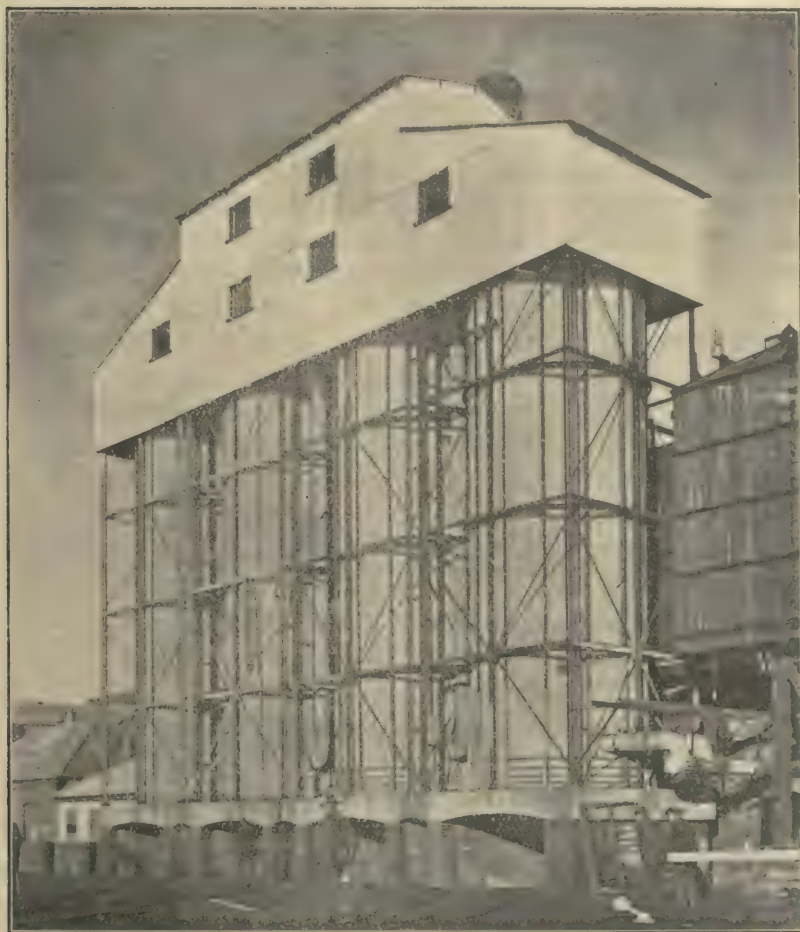
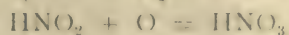


FIG. 151.

and is recuperated for the repetition of the oxidation reaction. These changes are not instantaneous, but require time, particularly the conversion of the nitric oxide. In these circumstances it is advisable to arrange the process to accord with the reactions.

The reduction of the nitrous acid is carried out in the former towers, and the oxidation of the nitric oxide is brought about in the latter towers.

The method of circulation of the process acid in the whole system is in consequence based on these reactions, that is, the acid from the latter towers is passed down the former towers and *vice versa*, thus if the installation contains six towers as shown in the accompanying photograph (Fig. 150), the arrangement for the circulation of the process acid is: No. 1 tower receives the nitrous process acid from No. 6 tower, No. 2 tower receives that from No. 5 tower, No. 3 receives that from No. 4. The sulphuric acid as made in towers Nos. 1, 2, 3, 4 and 5 is run into a central tank, where together with the acid from No. 6 it is raised and passed down No. 1 tower for denitrification and afterwards down the Glover tower for concentration. The process acid in the three former towers is cooled as necessary by means of leaden coolers.

The movement of the gas in the installation is effected by a suction fan placed between the towers and the Gay-Lussac tower. The introduction of the nitric acid is effected in tower No. 2 by adding a carefully regulated supply automatically controlled into the bottom tank receiving process acid from No. 5 tower to supply No. 2. The process acid in use in the three latter towers is cooled to remove the excess of heat produced in drying the gas.

The proportionate productivity of acid in the towers is approximately as follows:—

| | | |
|--------|-----|--------------------|
| No. 1. | 20% | of the total make. |
| No. 2. | 30% | „ „ „ |
| No. 3. | 50% | „ „ „ |

At the exit of No. 3 tower all the sulphur dioxide is oxidized to the last trace, so that the latter three towers function as absorption towers for the recovery of nitrogen oxides.

Messrs. Carmichael and Guillaume state that in comparing the vertical chamber (or tower) system with any other ordinary chamber system of equal capacity of production, it is found that the total volume only amounts to about one-fifth, and it is therefore claimed that the capital cost of the vertical chamber system is very considerably lower, and may amount to only one-half of the outlay, considering the ground occupied—a very important matter in certain works. Following on this, the capital charges are greatly reduced, and the maintenance charges are also lowered very considerably. After four years intensive work at 4 cubic feet per pound of sulphur, we are informed that there is no sign of depreciation in the structural lead.

The plant shown in Fig. 150 is a complete installation on the tower system, the towers being packed with patent glass packing (Figs. 152 and 153), and the installation is stated to be capable of working as low as $1\frac{1}{2}$ cubic feet per pound of sulphur per 24 hours when working on pyrites. Fig. 151 shows an installation of towers for dealing with dilute cold sulphur dioxide from the bastard Mannheim process after half the original sulphur has been converted into oleum. It is stated that in these towers acid up to 130° Tw. or 140° Tw. can be made direct from gases containing only $2\frac{1}{2}\%$ sulphur dioxide.

The plant shown is built on the Guillaume system of steel framework construction, for which the following advantages are claimed:—

- (1) Danger by fire eliminated.
- (2) Great reduction in weight, requiring smaller foundations, as compared with timber structure.
- (3) Increase in constructional rigidity and solidity, as compared with timber framework, so that it is possible to build much higher chambers, therefore greater suitability for working with sprays, providing a considerable economy as compared with the use of steam in the chambers, consequently the manufacturing cost of acid is reduced to a minimum. Higher chambers means less ground space occupied.
- (4) Chambers all covered in with any kind of roofing—slate, metal, wood or fire-proof material. Chambers all boarded in with wood or metal or cement slabs or brick filling between steel framework.
- (5) Greater accessibility for renewal or repairs as compared with the old system of timber framework. All steel parts are standardized and easily replaceable, being bolted together, not rivetted. The leadwork is easily reached at any point, as the steel framework is much more open than timber structure. The steelwork is so designed that all parts are readily accessible for painting with bitumen or acid-resisting paint.
- (6) Steel framework provides for 25% better condensation than timber structure, and thus ensures a proportionate increase in output of acid per unit chamber space, causing a reduction in capital outlay on plant.
- (7) Steel framework avoids all nails for fastening lead to wood; it also avoids horizontal straps. The differential expansion co-efficients as between lead and steel, and lead and wood ensure a longer life for the lead and less wear and tear. In ordinary timber chambers it is the wood framework which destroys the lead and necessitates the frequent repairs; this is the experience of over 30 years practical observation.
- (8) All lead burning is done on the outside, except the chamber floors. This arrangement gives security against leakage. Ordinary repairs are thus avoided, and the lead burning can be guaranteed to outlive the chamber lead.
- (9) The chambers are completely sealed, so that there is no escape of acid or gas.
- (10) Steel

framework chambers can be erected much more quickly than timber structure chambers, and therefore reduce the erection costs. (11) Steel framework chambers can be dismantled and re-erected without loss of material, the steel always retaining its value, which is not the case with timber. It is therefore specially suitable for hot climates, timber being useless in resisting heat and moisture and insect life. (12) The chambers can be made in any shape or size.

Glass-Packed Vitriol Towers.—Figs. 152 and 153 show a Gay-Lussac tower in which the patent glass packing of Messrs.

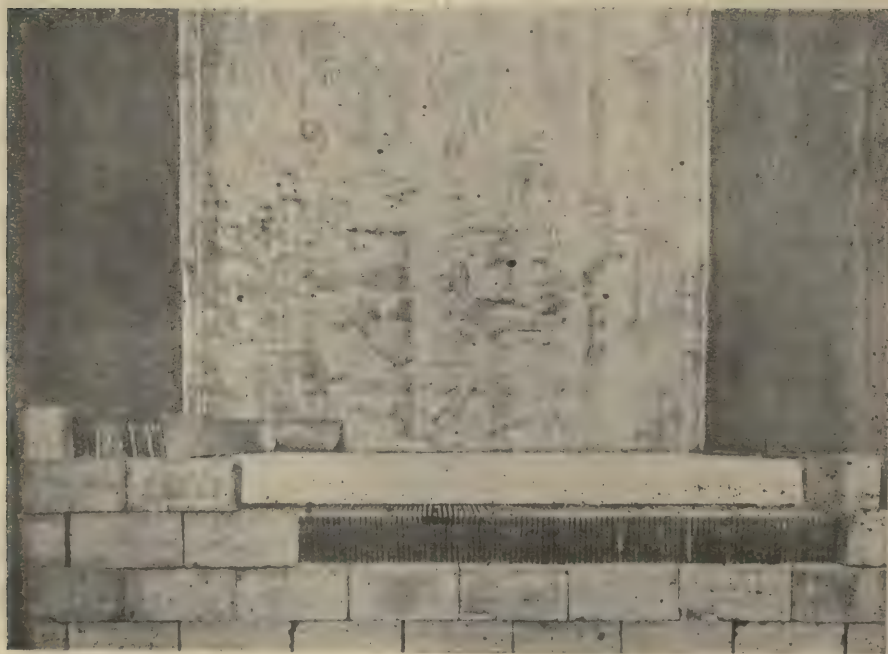


FIG. 152.—GLASS-PACKED GAY-LUSSAC TOWER IN COURSE OF ERECTION.

Carmichael and Guillaume is employed. Fig. 152 shows the Gay-Lussac in course of erection, and the method of fixing the glass packing; Fig. 153 is a top view of the tower.

It is claimed for this packing that it is the most efficient absorber for sulphuric acid work, providing three times more contact surface per unit than any other material, and that at the same time it offers the maximum free cross-sectional area for gas space, thus eliminat-

ing back pressure. It is further claimed that the packing material needs no renewal, that by its use the nitre consumption is lowered to an irreducible minimum, that it is unequalled as an intermediate tower, gives the lowest cubic co-efficient per unit of sulphur burnt, and that the output of vitriol is increased by 30% for 1% more cubic capacity;

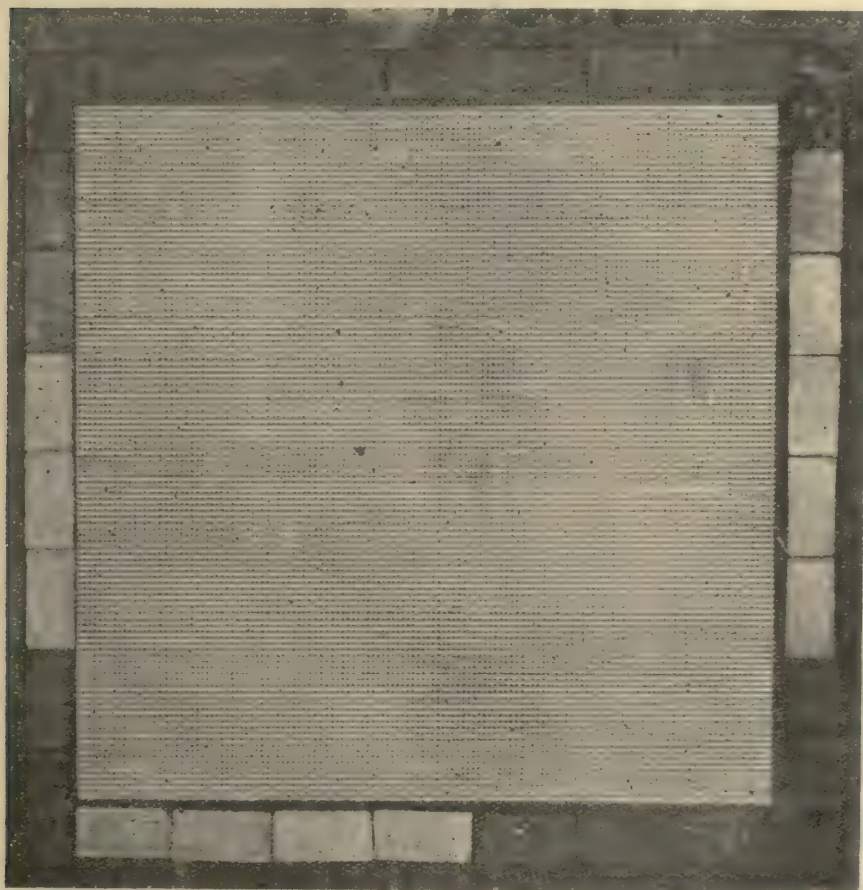


FIG. 153.—GLASS-PACKED GAY-LUSSAC TOWER: TOP VIEW.

moreover, that the glass-packed tower worked as a Gay-Lussac is the only type of tower which can be thoroughly washed down, as there are no horizontal surfaces of more than one-eighth of an inch in width, 96% of the contact surface being vertical surface; each course of packing is self-supported, and does not rest its weight on the course below it.

The following table show a comparison between the absorption effected in a Gay-Lussac tower 8ft. x 8ft. packed with bricks, and the same tower reduced in size to 6ft. x 6ft., and packed with glass. The figures represent tons of nitre absorbed per 24 hours:—

| Brick Tower 8ft. x 8ft. | | | Glass Tower 6ft. x 6ft. | | |
|-------------------------|-----|---------|-------------------------|-----|---------|
| 1910. | | | 1911. | | |
| September | ... | 0.36966 | September | ... | 0.51139 |
| October | ... | 0.34975 | October | ... | 0.56331 |
| November | ... | 0.37321 | November | ... | 0.62057 |
| December | ... | 0.34085 | December | ... | 0.58625 |
| January, 1911 | ... | 0.38706 | January, 1912 | ... | 0.55233 |

Comparative efficiency per cubic unit: Glass packing, 100; coke packing, 41; brick packing, 38.

Comparison between Glass Packing and Coke Packing for Gay-Lussac Towers.

New set consisting of 3 chambers (and glass packed contact tower) capacity 161,460 c. ft. Gay-Lussac tower, glass packed, 6ft. 0in. x 6ft. 0in. x 42ft.

Old set consisting of 5 chambers capacity 194,256 c. ft. and 2 Gay-Lussacs (coke packed when new), one 42ft. x 8ft. x 8ft., and the other 42ft. x 7ft. x 5ft.

| 1911/12 | Spent Oxide | Nitre. | % NaNO ₃ on Sulphur. | 1902/3 | Spent Oxide. | Nitre. | % NaNO ₃ on Sulphur. |
|---------|----------------|--------|---------------------------------------|--------|-----------------|--------|---------------------------------------|
| Dec. 1 | 52:10:0 | 16:2 | 3.9 | Dec. 5 | 73: 0:0 | 1:17:0 | 5.0 |
| " 8 | 52:10:0 | 12:2 | 2.3 | " 12 | 67: 0:0 | 1:14:2 | 5.1 |
| " 15 | 52:15:0 | 14:1 | 2.7 | " 19 | 75:10:0 | 1:11:2 | 4.1 |
| " 22 | 50: 7:0 | 1: 8:0 | 5.7 | " 26 | 66: 0:0 | 1: 9:0 | 4.4 |
| " 29 | 50:17:2 | 12:2 | 2.4 | | | | |
| Jan. 5 | 50:10:0 | 1: 0:2 | 4.0 | Jan. 2 | 62:10:0 | 1:12:2 | 5.1 |
| " 12 | 52:10:0 | 18:3 | 3.7 | " 9 | 73: 0:0 | 1:12:2 | 4.4 |
| " 19 | 52:10:0 | 15:1 | 2.9 | " 16 | 67: 0:0 | 1:13:0 | 4.9 |
| " 26 | 52:10:0 | 12:3 | 2.3 | " 23 | 69: 0:0 | 1:13:0 | 4.8 |
| | | | | " 30 | 62: 0:0 | 1:12:0 | 5.1 |
| Feb. 2 | 52: 5:0 | 17:2 | 3.5 | Feb. 6 | 68: 0:0 | 1:12:0 | 4.7 |
| " 9 | 52:10:0 | 16:0 | 3.0 | " 13 | 69:10:0 | 1:11:0 | 4.4 |
| " 16 | 52:10:0 | 13:0 | 2.4 | " 20 | 45: 0:0 | 18:0 | 4.0 |
| " 23 | 52:10:0 | 16:2 | 3.0 | " 27 | 45: 0:0 | 1: 2:0 | 4.9 |
| Mar. 1 | 52: 7:2 | 16:2 | 3.0 | Mar. 6 | 42:10:0 | 18:0 | 4.2 |
| " 8 | 52:10:0 | 14:3 | 2.6 | " 13 | 36:10:0 | 15:0 | 4.1 |
| " 15 | 52: 5:0 | 13:2 | 2.5 | " 20 | 38: 0:0 | 16:0 | 4.2 |
| " 22 | 52:10:0 | 13:2 | 2.4 | " 27 | 67: 0:0 | 2: 0:0 | 5.9 |
| " 29 | 48:17:2 | 14:3 | 2.9 | | | | |

Average, 52 tons. 3.0%

Average, 60 tons. 4.6%

Total cubic capacity of Gay-Lussac tower, 1512 c. ft.

Total capacity of Gay-Lussac towers, 4158 c. ft.

Glass.

Coke.

Comparative efficiency ... 4.2

1 per cubic unit.

The Guttman "Cell" System of Tower Packing.—Figs. 154, 155 and 156, 157, 158 and 159 show the system of packing towers by means of Guttman "cells." Fig. 154 shows the "B" type of cell, Fig. 155 the "C" type, Fig. 156 shows the gas distribution and the large proportion of free space, Fig. 157 shows the arrangement of the cells in a tower, Fig. 158 shows a system of four cells, and Fig. 159 shows the arrangement of cells in a circular tower. The following notes on the system have been furnished to the author by Messrs. Oscar Guttman and Sons.

(1) In comparing "Cells" with other types of filling material stress should be laid on the fact that in their passage through a cell tower the gases are continuously changing their velocity, thus giving a mixing action which brings fresh particles of gas into contact with the wetted surface. Filling material which presents the same cross-section to the gases in their upward passage through the tower does not stir the gases up in this way, and, in fact, the only possible way of obtaining this result is to cause a constant change of velocity. The mixing action of most kinds of filling material is usually the intermixing of portions of parallel streams of gas, which are diverted from one portion of the tower to another, without, however, producing sufficiently strong eddies to cause fresh particles of gas to come into contact with the wetted surfaces. It must be remembered that the velocity of the gases in a tower is usually very low, being generally something like 30-50 ft. per minute, and if nearly the same cross-section obtains throughout the tower, this is insufficient to cause strong eddies by impact against the surfaces or by mere deflection from them. In the cells the cross-section, when the gases pass through the slots, is reduced to about 15% of the area of the tower, thus giving the gases a velocity five or six times that obtaining at the widest part of the cell. This gives the good mixing action which is necessary for efficient reaction or absorption.

(2) Distribution of Gas and Liquid.—This is a most important matter, as unless the distribution both of gas and liquid is uniform, part of the volume of the tower is not utilised or only partly utilised. Most filling materials present their full section to the gases entering the tower, with the result that the gas has a tendency to take the shortest path, leaving the gas in other parts of the tower more or less stagnant. It is a matter of practical experience that the gas can be very uniformly distributed throughout a tower if it enters through a perforated plate with fairly small holes. The appearance in plan of the top layer of cells in a tower presents that of a perforated plate with slots instead of holes as the perforations. It follows that the distribution of gas in a cell tower

is very uniform and that the whole volume of the tower is utilised. It is equally important to obtain an equal distribution of liquid. In some types of filling material there is a tendency for drops to be formed, which fall from one layer to the next without wetting the surface of the material between the layers. With some filling materials the liquid has a tendency to collect in channels or to collect in one part of the tower. In the case of cells, the corrugations automatically arrange for correct distribution. If the distribution of liquid is uniform, each vertical row of cells practically acts as a small absorption tower of its own, the gases and liquid remaining in this vertical column. If, however, the neighbouring column should not be sufficiently wetted, there will be a certain amount of leakage between the layers of the cells, from one vertical column into the next, thus automatically adjusting unequal distribution in the first instance. It is sometimes urged that if the cells should not be placed truly horizontally, there will be a tendency for the liquid to run off at one end of the cell. A practical trial on a sample cell will disprove this, and it will be found that the corrugations will hold the liquid by capillary attraction, even if the cell is tilted 5 degrees or more.

(3) It is desirable for many reasons that a filling material should retain a small quantity of liquid, instead of draining completely, but it is not desirable to retain a large volume, as this would reduce the "free space." It will be found that cells retain on their corrugations just the correct quantity of liquid for most purposes. No figure is available giving the exact quantity, but it is found that it is about $\frac{1}{2}$ pint per cb. ft., so that a tower 6 ft. diameter and 20 ft. high would retain about 35 gallons of liquid. The more modern method of irrigating or distributing the liquid over a tower is to do so by periodic flushing rather than by steady drip. These flushings are usually carried out at intervals of thirty to sixty seconds. At each flushing the liquid already on the filling is partly displaced or replaced. If the liquid travels too quickly down the tower the result is not so good as if the change is effected slowly. In most filling materials which drain themselves completely, the liquid will run from the top to the bottom of the tower in three or four seconds. In the case of cells it will be found that it takes from fifteen to thirty seconds, which is the most favourable time for the system of periodic flushing. The quantity of liquid retained by the cells will also be found to correspond approximately to the quantity of liquid flushed over the tower in each minute. It has been found by experience that the best quantity of liquid to be circulated over a cell tower is between $\frac{1}{2}$ and 1 gallon per minute per square foot of tower area, and that this varies with

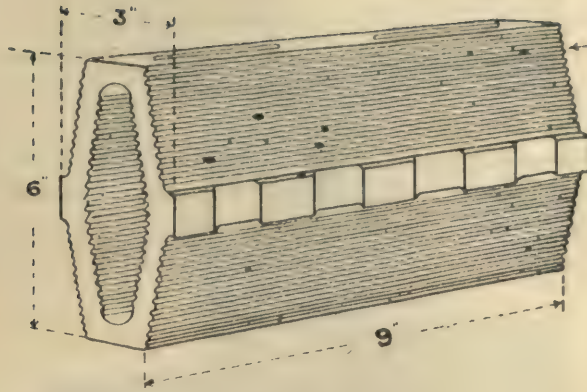


FIG. 154.

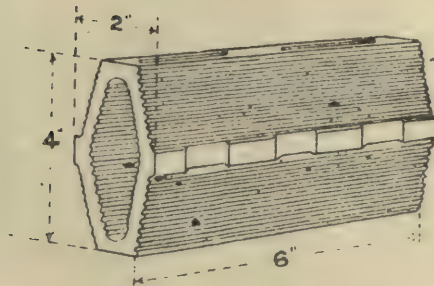


FIG. 155.

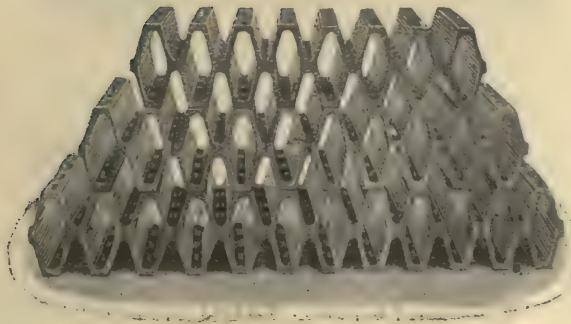


FIG. 156

GUTTMANN "CELL" TOWER PACKING.

the nature of the gases and liquid used. This refers to towers over which the same liquid is circulated continuously until it reaches a certain strength. For some purposes the liquid has only to be passed down the tower once, and in that case the rate of flow is usually much less.

(4) In the case of dusty gases, thick liquids or sludges, it is essential that the sides of the filling material should have as steep an angle as possible in order to prevent accumulation of sediment or choking up in the tower. This condition is fulfilled by cells, which have been in use with thick milk of lime and heavy anthracene oil without any signs

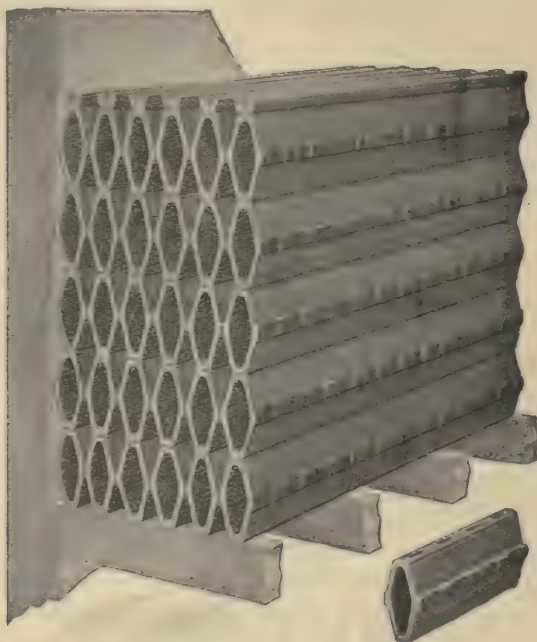


FIG. 157.—GUTTMANN "CELL" TOWER PACKING.

of choking. Should sediment collect, the steep sides enable flushing or cleaning out to be effected quickly and efficiently.

(5) In most cases it may be assumed that a cell tower will do the same work as a coke tower of about six times its volume, but this would not apply for very special purposes. As compared with other types of filling material, the efficiency of cells would, other things being equal, be proportional to their free space. Thus, in comparing cells having a free space of 74% with another filling material having a free space of 50%, a cell tower would only require 50 cb. ft. of cells to do the same work as 74 cb. ft. of the other filling material. The result of this superiority is

the possibility of using a smaller tower, and the extra cost of the packing (if any) is more than compensated for by the saving in the cost of the tower, quite apart from annual savings in regard to pumping, etc.

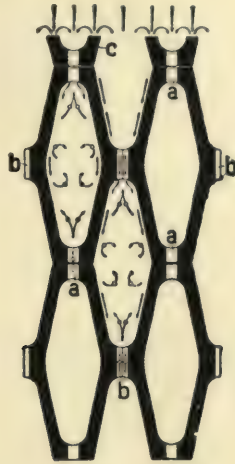


FIG. 158.

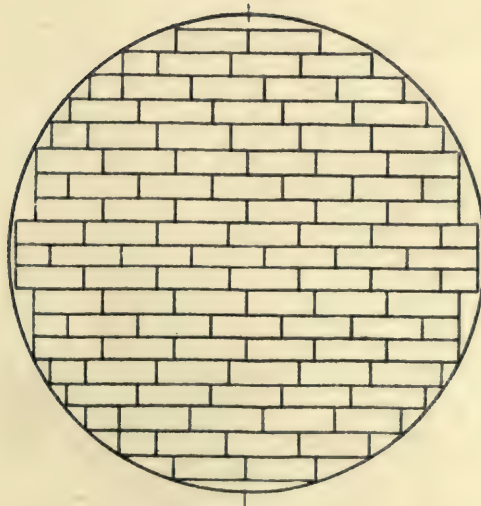


FIG. 159.

(6) Cells can be readily packed both in rectangular and circular towers, and no special skill is required in stacking them, as they are merely built up and "bonded" like ordinary brickwork. It should be

noted, however, that the dimensions of a rectangular tower should always, if possible, be a multiple of the length or half length of a cell. Thus for cells type B a tower should be a multiple of 9 in. or $4\frac{1}{2}$ in., and for type C a multiple of 6 in. or 3 in. These dimensions will be found to fit in very well with the sizes of bricks which are usually used. Rectangular towers are an advantage in many cases, because several towers can then be conveniently joined into one structure, a side leakage through the internal partition walls being of little consequence. A saving in the cost of the towers results from this.

(7) With cells there is no bursting pressure on the tower walls as would be the case with coke, balls or the like.

Crushing Tests.—A single cell was placed on a board and 56 lb. weights were placed on another board on top of the cell.

One cell type B, 9 in. long, withstood a crushing weight of one ton, and the crushing weight was only slightly reduced when a cell was tested which had been purposely selected as having some hair and other cracks. This corresponds to a stacked height of 260 ft.

A single cell type C, 6 in. long, withstood 11 cwt. corresponding to a stacked height of 550 ft.

Weights.—20 cells type B, 9 in. long, weighed $84\frac{1}{2}$ lbs. As there are 10 and two-thirds of these cells per cb. ft., cell filling type B weighs approximately 45 lbs. per cb. ft.

40 cells type C, 6 in. long, weighed $55\frac{1}{2}$ lbs., and as there are 36 of these per cb. ft., the weight of cell filling type C is approximately 49 lbs. per cb. ft. as at present manufactured.

Fig. 154 gives the dimensions of cells type B, which are used for the general run of chemical plant such as Gay-Lussac towers, the upper part of Glover towers, etc. They have also proved particularly efficient in towers for the manufacture of nitric acid by the arc and Ostwald processes, in which a high "free space" together with efficient mixing is practically of paramount importance, it being only necessary to provide a relatively moderate amount of scrubbing surface. Fig. 155 gives the dimensions of cells type C, which have the largest amount of scrubbing surface (34 sq. ft. per cb. ft.), and these are used chiefly for smaller absorption towers, such as in nitric acid plant, sulphur dioxide absorption and the like. A larger size type A is also made for the lower part of Glover towers, for bacterial treatment of sewage, and for other special purposes. Fig. 159 shows the method of stacking cells in circular towers, the spaces between the cells and the walls of the tower being filled in with broken ware.

(9) All sizes of cells have a free space of 74%, and this is most important, as, other things being equal, the higher the free space the smaller the volume of the tower may be for a given volume of gas per minute.

Cells type B have a scrubbing surface of 22.5 sq. ft. per cb. ft. (67.5 sq. yards per cb. yard) and approximately 11 cells go to the cb. ft. Cells type C possess 34 sq. ft. of scrubbing surface per cb. ft. (102 sq. yards per cb. yard) and 36 cells go to the cb. ft.

Very large quantities of these cells are in satisfactory use at the present time; they are now made of Accrington acid-proof brick, and as regards price they compare very favourably with any other type of filling material, having regard to their high practical efficiency and scientific design.

The Mills-Packard Vitriol Chamber.—The following notes on the system have been supplied to the author by Messrs. Edward Packard and Co., of Ipswich. Fig. 160 shows an installation of Mills-Packard Chambers.

The chief difference between the ordinary chamber system and the Mills-Packard system lies in the shape of the chambers, which, in the latter type, are in the form of truncated cones externally cooled with water. At the top of the outside of the lead shell is placed a gutter, from which the water is distributed so as to obtain a complete cooling of the whole surface. At a certain distance below this gutter is a further gutter which re-collects the water which has travelled over the surface of the lead and forms a reservoir from which the water is re-distributed over the section of the surface immediately below. Below this again at a certain distance is a further gutter re-collecting the water, and so on at certain intervals until the chamber dish is reached. This arrangement allows the height of the chamber to be divided up into a number of sections, each of which is re-cooled independently of the other, that is to say, a complete re-distribution of the cooling water is made at intervals, so as to obtain a far more complete covering of the surface than would be possible if the moisture were allowed to run unchecked from top to bottom. It is claimed that such a process permits of a very high production, as intensive condensation is due to the re-cooling of gases by the cooling water and assures a very energetic mixing of the gases. The quantity of water employed on the outside of the chamber is increased or diminished according to the intensity of production and the temperature of the atmosphere. From the fact of the conical shape

of these chambers, the gases entering at the bottom and travelling to the top, coming in contact with more intense cooling at the top due to the tapering of the chambers, are subjected to increased cooling at the most intense point in the reaction.

The Mills-Packard chambers are constructed without any covering, in accordance with the usual custom in England, but there is no difficulty about housing them in, as is usual in Continental works. In fact, it would probably be advisable where great changes in temperature are encountered, and this is being carried out in a plant now under erection at the time of writing.

It had been often noticed that where lead chambers are exposed to the atmosphere, the chambers are filled with nitrous gases after a shower of rain on a hot day, or during a cool night following a hot day, and the patentee desired to obtain these conditions continuously. The results obtained have not only justified the experiment in this direction, but other advantages of water-cooling have become apparent. So intense does the reaction become, owing to the rapid cooling of the gases, that the chamber space can be reduced to between 3 and 4 cubic feet per pound of sulphur. In spite, however, of this intensive working, the wear and tear on the chambers is actually reduced by keeping the lead shell at a constantly low temperature.

It is claimed for the Mills-Packard system that it has the advantage over the Opl tower system that no circulation of acid is necessary.

With intensive working on ordinary lead chambers a much more rapid deterioration of the lead is experienced owing to the high temperatures. This is obviated in the Mills-Packard chambers by the external water cooling, by which the temperature of the lead shell is kept down to within a few degrees of the temperature of the cooling water. In practice in England, the temperature of the lead of the first chambers varies from 18° to 30° C. (66° to 86° F.) which explains the very low wear and tear. In actual experiment in one case the loss of weight after four years working was less than 1% of the original weight of the lead. In another case the loss was practically *nil*, and it is unnecessary to call attention to the low cost of maintenance at such temperatures as those mentioned above.

With these chambers acidity of the exit gases from the Gay-Lussac towers is unusually low, and has seldom been known to exceed 2 grains per cubic foot, and with regular work varies between 0.4 and 1 grain.

The daily control of the process is claimed to be not more

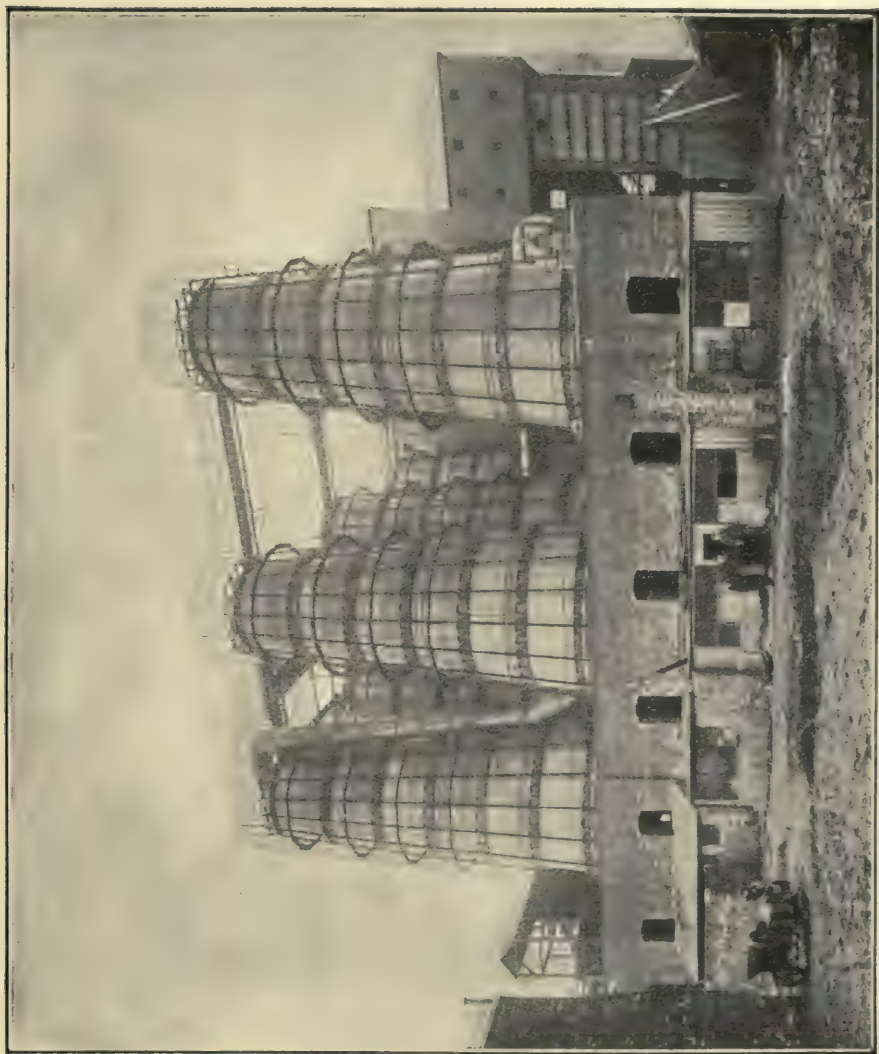


FIG. 160.—INSTALLATION OF MILLS-PACKARD VITRIOL CHAMBERS.

difficult than with the old rectangular chambers, as any irregularity in working is more easily corrected.

Below are given particulars of four sets of Mills-Packard chambers; the first working on spent oxide, the second on zinc blende roasted in a Delplace furnace, and the third and fourth on pyrites.

(1.)—MATERIAL, SPENT OXIDE.

Production, 19 kilos to 60° Bé acid per cubic metre = 3.3 c. ft. chamber space per lb. sulphur charged per 24 hours.

% Nitrate on sulphur burnt = 3.0.

| | Temperature. | Strength (Drip). |
|--|--------------|------------------|
| First chamber ... | 79° C. | 125° Tw. |
| Second chamber ... | 47° C. | 118° Tw. |
| Exit gases, 0.3 grains SO ₃ per c.ft. | | |
| Maximum temperature of lead, | | |
| first chamber ... | 27° C. | |

(2.)—MATERIAL, ZINC BLENDE.

Production 14.29 kilos per cubic metre (60° Bé acid) equals 4.4 c.ft. chamber space per lb. sulphur.

% Nitrate on sulphur burnt = 2.0.

| | Temperature. | Strength (Drip). |
|--|--------------|------------------|
| First chamber ... | 49° C. | 127° Tw. |
| Second chamber ... | 54° C. | 125° Tw. |
| Third chamber ... | 35° C. | 97° Tw. |
| Exit gases, 0.7 grain SO ₃ per c.ft. | | |
| Maximum temperature of lead, first chamber, 19° C. | | |

(3.)—MATERIAL, PYRITES.

Production 21.88 kilos 60° Bé acid per cubic metre = 2.87 c.ft. chamber space per lb. of sulphur.

% Nitrate on sulphur burnt = 2.75.

| | Temperature. | Strength (Drip). |
|--|--------------|------------------|
| First chamber ... | 70° C. | 132° Tw. |
| Second chamber ... | 54° C. | 130° Tw. |
| Third chamber ... | 36° C. | 108° Tw. |
| Exit gases under 1 grain SO ₃ per c.ft. | | |
| Maximum temperature of lead, first chamber 24° C. | | |

(4.)—MATERIAL, PYRITES (roasted on Herreshoff furnaces).

Production 17.28 kilos per cubic metre 60° Bé acid = 3.6 c.ft. chamber space per lb. of sulphur charged per 24 hours.

Exit gases 1.1 grains SO₃ per c.ft.

CHAMBER TEMPERATURES AND DRIPS.

| Chamber. | Temperature° C. | | | | | | Drip ° Tw. |
|---------------|-----------------|-----|-----|----|-----|-----|------------|
| No. A1 | ... | ... | ... | 73 | ... | ... | 132 |
| A2 | ... | ... | ... | 73 | ... | ... | 131 |
| B1 | ... | ... | ... | 57 | ... | ... | 130 |
| B2 | ... | ... | ... | 54 | ... | ... | 131 |
| C | ... | ... | ... | 49 | ... | ... | 100 |
| D | ... | ... | ... | 34 | ... | ... | 94 |

Maximum temperature of lead chambers A1 and A2 = 24° C.

In October, 1918, sixty-three Mills-Packard chambers were either working or in course of construction in England and the Colonies, and with two exceptions the standard-sized chamber of 7,330 c. ft. has been adopted. In the other cases, chambers of 11,000 c. ft. and 18,000 c. ft. have been erected. Up to the present the maximum size compatible with good results has not been determined, but it is evident that the diameter of the chamber must not be so great as to reduce the effects of the water-cooling.

Mills-Packard chambers can be employed either in complete units of several chambers in a set or in conjunction with rectangular chambers, when they take the place of intermediate towers, than which they are less costly and more simple to construct.

The chambers in reality resemble towers, but are claimed to possess none of the disadvantages generally associated with towers.

In conclusion, it is claimed for these chambers that they possess the following advantages.—

- (1.) High production with low nitre consumption.
- (2.) Low wear and tear on lead.
- (3.) First cost of construction per ton of acid reduced to a minimum.
- (4.) Very small space required.
- (5.) Simplicity of construction.

The Moritz Burner.—The Moritz burner of Messrs. Simon Carves, Ltd., of Manchester, is shown in Fig. 161, and is a good example of the mechanical pyrites burner. In a communication to the author from the makers it is pointed out that in Moritz burners which are designed for working on pyrites fines there is entire absence of overheating, large capacity for overloads, air drying of the green ore, and carefully studied accessibility, which allow for the easy renewal of the arms or rabblers when required. This burner is of the type originally designed by McDougall, but the two difficulties (overheating

and dust) that rendered McDougall's device unworkable have in this case been entirely overcome. Owing to the high temperature existing in the interior of the furnace, the centre shaft and arms tend to wear out prematurely. The natural step then is to introduce cooling medium into these parts. By some inventors water was used, but it was found that amongst many insuperable difficulties, incrustation occurred inside the arms and shaft. Further, water turned out to be an insufficiently elastic agent from a thermal point of view, and owing to

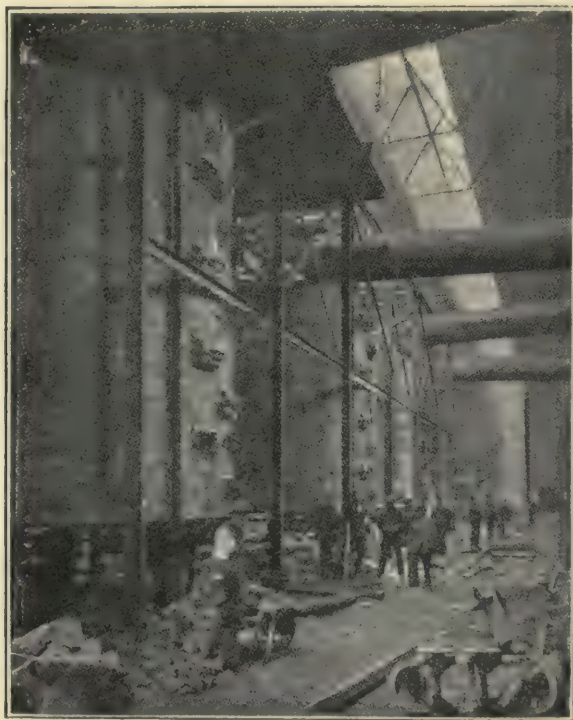


FIG. 161.—INSTALLATION OF MORITZ PYRITES BURNERS.

the formation of steam locks the circulation was impeded where cooling was most required, and in case of repairs or breakage of the arms, the inevitable leakage of water was constantly a source of risk, danger and damage.

The Moritz burner consists of a large sheet iron cylinder lined with firebrick and containing light circular firebrick hearths, built with a slight fall from the centre to the circumference; thus they are self-supporting by reason of their arch-like section. Through the centre

of these hearths there rises the vertical rotating centre shaft, to which are attached the sixteen arms, two for each of the eight shelves, each arm having a number of hanging blades or teeth, set at an angle of 45° to the axis of the arms, and so arranged as to overlap each other in their circumferential path, thus ploughing the pyrites fines outwards to the circumference of the burner, or inwards to the central shaft in each alternate shelf. The pyrites are introduced at the outer edge of the top hearth by a piston feed, operated by a large cam, of variable travel, driven by the top portion of the central shaft. The arms of this hearth are equipped with teeth, so shaped that the ore is raked to the centre, where it falls through an annular opening on to the second hearth. Here the teeth are set at such an angle that the ore is carried outwards to the circumference, where it glides down to the third hearth through a series of spiral openings, specially formed to minimise the creation of dust. Thus the ore travels down the burner until it is finally automatically discharged, quite cold, at the circumference of the bottom hearth into the waiting truck. The air required for combustion is admitted round this hearth, and rises upwards together with the resulting gases in a direction opposite to that in which the ore is descending. Air is supplied by a small fan to cool the shaft and arms, and there is claimed an important improvement over other burners of the air-cooled type, in that the centre cast-iron shaft is formed of two concentric passages, the arms being connected to both passages by a special self-locking joint. Only the arms in the four top hearths are air-cooled. The course of the air is as follows: At the bottom of the burners it is admitted into the outer section of the shaft, which is thus most effectively cooled. From here it passes to the top end of each arm (the arms being divided into two compartments by a longitudinal partition, which also serves as a strengthening rib) and then backwards into the central portion of the cast-iron vertical shaft. It will thus be seen that the air heated by the arms is kept away from that portion of the central shaft exposed to the burning ore, the outer portion of the central shaft being supplied with cold air as it enters, and thus overheating and sulphuration of the iron are entirely prevented. Two arms are generally used in each hearth, but four may be provided in exceptional cases. The hot air from the central cast-iron shaft passes into a flat box fixed about 6ft. above the top of the burner, and finally into the atmosphere, the roof of this box forming a drying floor for a twenty-four hours supply of pyrites. Free expansion of the centre shaft is permitted by a gravel

seal at the top, and any permanent increase in length or settlement of the arches can be compensated for without disturbing the rest of the burner, by the removal of special cast iron packing rings at the base of the shaft, the operation requiring only a few minutes. The burners are made in 3, 5, and 8-ton sizes, this being a 24-hours rating. There are, however, burners at present at work producing over 80% in excess of their rated capacity. When working with cupreous pyrites at least 80% of the copper is rendered directly soluble without chlorination. The power required to drive the moving parts of the burner is exceedingly small, being in the neighbourhood of five-eighths of 1 H.P. The feed and discharge are, as explained above, entirely automatic, all that is necessary being to load the pyrites into a hopper from the drying floor,



FIG. 162.—GROUP OF "CERATHERM" APPARATUS BY GUTHRIE AND CO.

which is capable of easily holding sufficient for 24 hours working. The special features claimed for the Moritz mechanical pyrites burner are:— (a) Exceptionally high temperature of the gases leaving the burner. The cinders leaving the burner are at practically atmospheric temperature. (b) Complete de-sulphurisation of the pyrites to within 2% of the total expungeable sulphur. (c) Efficient air-cooling of easily removable and interchangeable arms, hence long life. (d) Safety adjustable drive-release-gear, with loud sounding gong, operating in case of "rabbles" or other obstruction to arms, thus breakage and damage prevented. (e) Power required, $\frac{1}{4}$ H.P. per 8-ton burner, no friction, and hence no wear. (f) Exceptional elasticity, 40% under or overload easily possible without material loss of efficiency in burning. (g) Small ground space, for small or large units.

"Ceratherm" Sulphuric Acid Plant.—Fig. 162 shows a group of fittings for sulphuric acid concentration plant in "Ceratherm" by Messrs. Guthrie and Co., of Accrington, Lancs. This non-corrosive material, which is now in extensive use, has the properties of a ceramic substance like porcelain as regards its resistance to acids, and possesses valuable thermal properties in addition. In a communication to the author, Messrs. Guthrie and Co. point out that there has long been

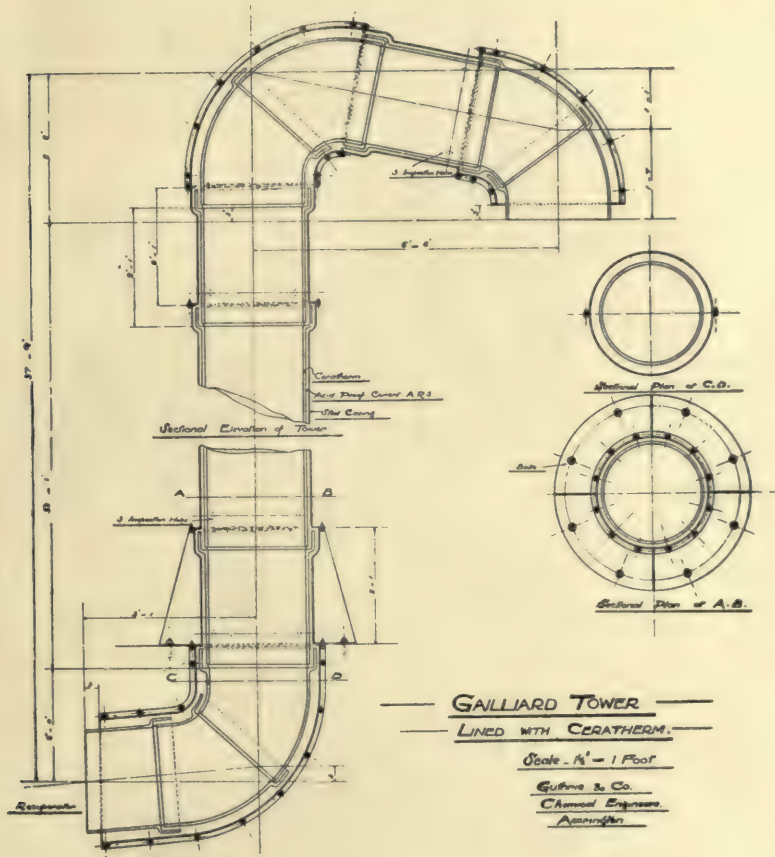


FIG. 163.—GALLIARD TOWER CONNECTOR, LINED WITH "CERATHERM," BY GUTHRIE AND CO.

a need for a material which would combine the valuable properties of the inertness of porcelain to chemicals and acids with the capacity to withstand violent changes of temperature similar to fused silica, but with a higher thermal conductivity and a higher tensile strength than substances previously available; that Ceratherm is an acid-ware

and is absolutely acid proof; small articles of this material may be heated to a red heat and plunged into cold water without cracking; that the thermal conductivity and emissivity are very much higher than that of either fused silica or stoneware; that it is not fragile and not so liable to be broken as stoneware and fused silica; that it is light in weight, its specific gravity being approximately 2.95, which gives it a marked advantage over acid-resisting metals; and that it is of homogeneous quality and not affected by hydrochloric acid and nitrosyl chloride, etc. Ceratherm has been used on cascade concentration plant very satisfactorily; the tensile strength of the material makes it less liable to be broken than a silica or porcelain basin. In cases where the recovery of waste acid from inflammable and explosive materials is necessary, and where freedom from metallic contamination is essential, Ceratherm is specially suitable. Basins are made up to 2ft. diameter.

A large number of Ceratherm calottes and covers are in use in Kessler plant, and have given very satisfactory results, due to their freedom from breakage through change of temperature. Fig. 162 shows Ceratherm cascade basins (2ft. and 1ft.) Kessler covers and calottes, furnace pipes, hot flue bend, large double inlet pipes, distributing plate, Hart's headers (for nitric acid plant), "S" condensing pipes, and connector to air-cooled prism (referred to in Chapter V).

In the Gaillard process, one of the principal troubles is in the connection between the recuperator and the main tower. Fig. 163 shows a steel-armoured Ceratherm connector, which contains a Ceratherm internal tower coated with a special type of acid-resisting cement and held together by a suitable shield. The steel shield and acid-resisting cement are stated to be not necessary, but used as an expedient means of substantially setting the Ceratherm tower; such towers are being put up without the steel armouring, and supported only by wooden framework.

CHAPTER V.

NITRIC ACID PLANT.

It is hardly to be expected that a comparatively simple process such as the production of nitric acid from sodium nitrate by direct distillation can be susceptible of drastic change. Such changes in the production of nitric acid are to be looked for in connection with the various catalytic processes which are at present in this country only in the experimental stage. Attempts are being made to perfect the continuous method of distillation, but so far the intermittent process holds the field. The stress of war conditions has, of course, enabled every type of distillation plant to be thoroughly tried, with consequent modification and improvement.

With the outbreak of war in 1914 began a feverish development of nitric acid manufacture. Every consideration gave way to speed of production. Every firm that produced anything in the nature of plant and parts for making nitric acid was pressed to supply what it could. Thus in the course of a few months almost every possible type of nitric acid plant that could be found on the market had its opportunity. The purpose of these notes is to record a few of the changes that war conditions have wrought in this industry and to comment on the improvement that has been made.

Process.—No change has taken place in the main portion of the process. The present general procedure is somewhat as follows:—

The nitrate is brought to the charging platform in weighed quantities, and while one charge is distilling, the next charge is drying. The drying should not be carried too far, as the nitrate may fire when charged into the pot not properly cooled. If the charge is, say, 2 tons

of nitrate of soda reckoned as 96% NaNO_3 , the quantity of sulphuric acid of 168°Tw. (95 to 96% H_2SO_4) is about 8% in excess of the theoretical quantity required to produce the bisulphate, say about $2\frac{1}{4}$ tons. In practice the average strength of C.O.V. is only 92%. Heating is conducted slowly at first, about an hour intervening before any vapours are seen in the observation lantern. Modern practice demands no temperature control, but a reliable thermometer gives a good idea of the progress of distillation. There should be one thermometer depending in the liquid mass in the still and another near the front end of the condenser. In some plants the thermometer is fixed in the still arm above the retort. Danger of frothing or violent reaction occurs at two points which can be determined readily by observation. In the Guttman plant the first of these danger points occurs when the second thermometer registers 65°C. ; the second point when this temperature rises to 100°C. Single unit plants are generally provided with two condensing towers, the final tower being supplied with water circulated by means of a montejus or air lift till the acid formed registers 71° to 72°Tw. Steam may be also admitted. Where sulphuric acid chambers are available the uncondensed vapours issuing from the first catch towers are delivered to these chambers.

Distillation lasts from eight to sixteen hours, depending on the size of condenser and the design of retorts, etc. Twelve hours is a good average. Towards the end weak acid is produced of 71 to 73°Tw. (1.360 sp.gr.) and the fire is hotter than at the commencement of the distillation. This weak acid is collected separately, and together with the weak acid produced in the absorbing towers may be added to the next charge. In this way and in suitable plant practically all the acid may be produced as strong 1.5 sp. gr. The strong acid is collected and stored in suitable lead tanks or containers. Where the strong acid is required white and free from brown fumes a small current of air, usually warmed, is blown in a separate vessel termed a bleacher, or in the condensing pipes. In some plants the bleacher is placed between the still and condensing system. In others, as for instance, the Guttman system, the bleaching vessel is at the end of the plant.

After the charge is finished the bisulphate is run out, and the fire and flue cleaning doors are opened to cool the pot, which usually takes about two hours. The pot may then be re-charged. In the Guttman plant the foul gases, consisting chiefly of chlorine, that come over on first heating of the retort, are quickly swept through the system by admitting warm air through a separate cock in direct connection with the retort. The same cock may also be used at the end of the

charge to admit air to cool the pot. In other systems this refinement is not fitted. Both the outlet plug and the nitrate charging opening are generally luted with fireclay.

It is usual in modern plants to control the temperature gradient by the film cooling system. This is done by water spray assisted by various spreading devices. An extreme type is a canvas fabric hung over the condensing pipes. The placing of pipes in tanks is not recommended

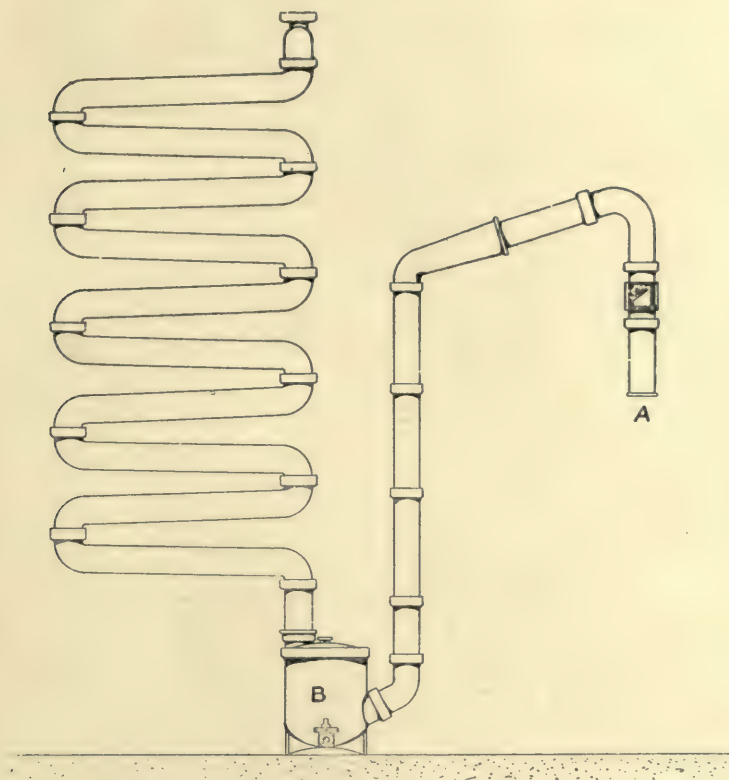


FIG. 164.

on account of the difficulty of removing a cracked pipe. As will be mentioned later, a modern type of condensing plant consists of pipes each having its own cooling jacket. Where film cooling is adopted, care is taken to keep the outside of the pipes clean, and free from grease and scale, as otherwise the film does not cover completely the external surface. When the still is being cooled or charged it is not desirable completely to shut off the cooling water, because the drying of the surface

disturbs the even flow of the film. About every six months or so the retort should be entered and examined for corrosion, especially those parts in direct contact with the fire. Flaws in the casting which are hidden when the retort is fixed appear sooner or later as local areas of corrosion.

The sulphuric acid, being strong, is stored in steel vessels placed at such a height that gravity flow may be used. This is usually more convenient than blowing up the acid for each charge from an egg or montejus.

Regarding buildings, it is sufficient to say that ventilation must be provided, but care should be taken to prevent draughts blowing round earthenware, or even some makes of silicon alloys.

Better arrangements are now made for the removal of the nitre cake. The usual arrangement is a row of large cast-iron dishes on brickwork piers to allow of air cooling. These are often in the open, but it is preferable to cover with a light shed, for moisture soon converts the solid cake into a corrosive mess. Some plants are fitted with bogey tip wagons capable of being run under the retort outlet. The block of cake is removed by hoop-iron hooks cast in the liquid mass.

The Retort and Setting.—Of the many types of retorts that have been designed for nitric acid distillation, the vertical retort is now the usual form. The two-ton size is usually 10 ft. deep by 7 ft. diameter, and is made in two sections. The smaller size is usually made in one piece. Some chemical engineers prefer the retorts built up in sections in all cases. The Guttman retort, for instance, which is made in two standard sizes for $2\frac{1}{2}$ and $3\frac{1}{2}$ tons nitre charge respectively, has a lower section with spout, another section fitted into a socket, and a cover of the same diameter as the pot, having upon it all outlet connections and charging openings. The mixture of acid and nitre does not fill the lowest section, and there is therefore no danger from leakage. The bottom may be made thicker than the middle section and cover, thus saving in first cost and replacement. Barring accidents, this casting should last 3 to 5 years. The joints of the retort sections are usually made with rust cement. Sometimes ordinary fireclay is used, but is not to be recommended. The Valentiner still is also much used, and is cast in the form of a kettle, all in one piece, the opening at the top being smaller than the diameter of the still. There are yet a few of the old-time cylinder type of still in existence. This form is not to

be recommended, though it possesses advantages in the way of easy access for cleaning. The cylindrical form is moreover easier to cast sound than the Valentiner shape. British Dyes, Ltd., have, however, installed a number of horizontal retorts working under atmospheric pressure*. The best mixture of iron is such as to ensure a casting with a tough interior and an acid-resisting skin. Orders for nitric acid retorts should be given to firms having special experience in this line.

Nothing fundamentally new is to be noted in the way of settings. The Guttman type of setting has been almost universally adopted. Provided that the fierce heating of the bottom of the retort is prevented by some form of protecting arch, and the flues so arranged that the fire gases pass easily round the retort, no difficulties can reasonably be expected. The cover of the pot is to be hot enough to prevent condensation of the nitric acid. Coke is the usual fuel employed, chiefly on account of its cheapness, freedom from smoke, and absence of flame. In a case of benches of retorts of, say, eight and upwards, a number of firms have installed gas firing, the gas being provided in one of the usual forms of producer, as, for instance, the Mond or Kerpeley. The control and management of the temperature of gas firing is much easier than that of a coke fired furnace. It entails less labour and the man is free then to perform minor subsidiary tasks that lie about the place. There are no ashes, no dust, and under careful supervision gas firing is a distinct advance.

The chief differences to be noted in the many plants which are now appearing is in the condensing system. Whereas in the old days the only material that was available was stoneware, there are now several forms of high silicon alloys and the well-known fused silica ware. The latter ware has developed enormously as a material where acids of all kinds are conveyed through pipe-work and in small vessels. There are also distinct differences in design. These differences are mainly due to the properties of the new materials and the conditions of their manufacture. Stoneware, of course, made in the usual potters' way, should be made thin and in small pieces, and with socket joints. Unless standardised it requires a lengthy period for its manufacture, owing to the need for slow drying. Silica ware is made in the electric furnace, and hence quickly. It cannot be made in large pieces, but, on the other hand, it can be had in fairly lengthy coils. The designs in silicon iron alloys are directed chiefly towards ease in manufacture in the foundry—in fact, the difficulties in the foundry determine practically the form which these alloys should take. At

* *Chemical Trade Journal*, June 8th, 1918, p. 463.

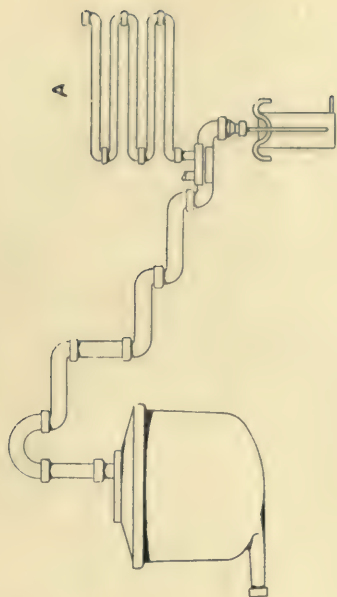


FIG. 166.



FIG. 167.

first, because the design of these alloys was in the hands of the foundry manager, the forms which articles of this material took followed all ordinary cast iron ware. Thus we had the flanged pipe joint. In practice the silicon iron alloys behaved much more like a glass or a very brittle stoneware than ordinary iron. The flanges cracked and difficulties were found in the making of joints by means of ordinary wrought iron bolts. Slight leaks of acid corroded the bolts. Very often at the flange the metal was porous, and the acid "weeping" through came in contact with the bolts. Many forms of plant were condemned on this point alone. Iron alloy pipes are usually supplied now with socket joints connected up, just like any other stoneware or silica ware pipes. Another change in the design of the plant is the development of what is now termed the reflux design. In this plant the gases are condensed in a series of pipes of a "S" form, and are connected to a vessel into which the condensed acid runs. The uncondensed vapours from this vessel rise up through another series of pipes, or very often a coil, the acid condensing in this running back into the previously mentioned vessel. The upper end of the coil or reflux pipe is connected to the condensing tower system. The development in the design of the condensing part of the nitric acid plant can best be seen by a study of the chief representative types.

Fig. 164 is a well-known form of condensing plant in use in a large Scotch works, and is the result of very many years experience with silicon iron alloys. A glance at the illustration would convey to any chemical engineer that it has got down to almost the last stage of simplicity. The pipe marked "A" is the uptake from the retort. The gases pass up this over the joint and down direct to the bleacher "B." Any uncondensed gases then rise up through a series of socketted "S" pipes, ranged vertically and supported by some form of iron or preferably wooden framework. As this plant is usually put up in large groups, a set of condensing towers is erected to condense the vapours from a number of retorts. This is done, of course, in the usual manner. To assist the condensation a perforated pipe is suspended over the uppermost "S" pipe and a trickle of water allowed to fall from pipe to pipe, covering each with a film of water, and aiding by its evaporation the condensation of the nitric acid vapours inside. Early forms of this plant were made with flanged joints, and as has already been stated, the flanged joints have been discarded in view of the difficulties of keeping the joints tight. Here the claims of the manufacturer have overcome the prejudices of the foundry. The bleaching in this instance is done by bubbling a small stream of

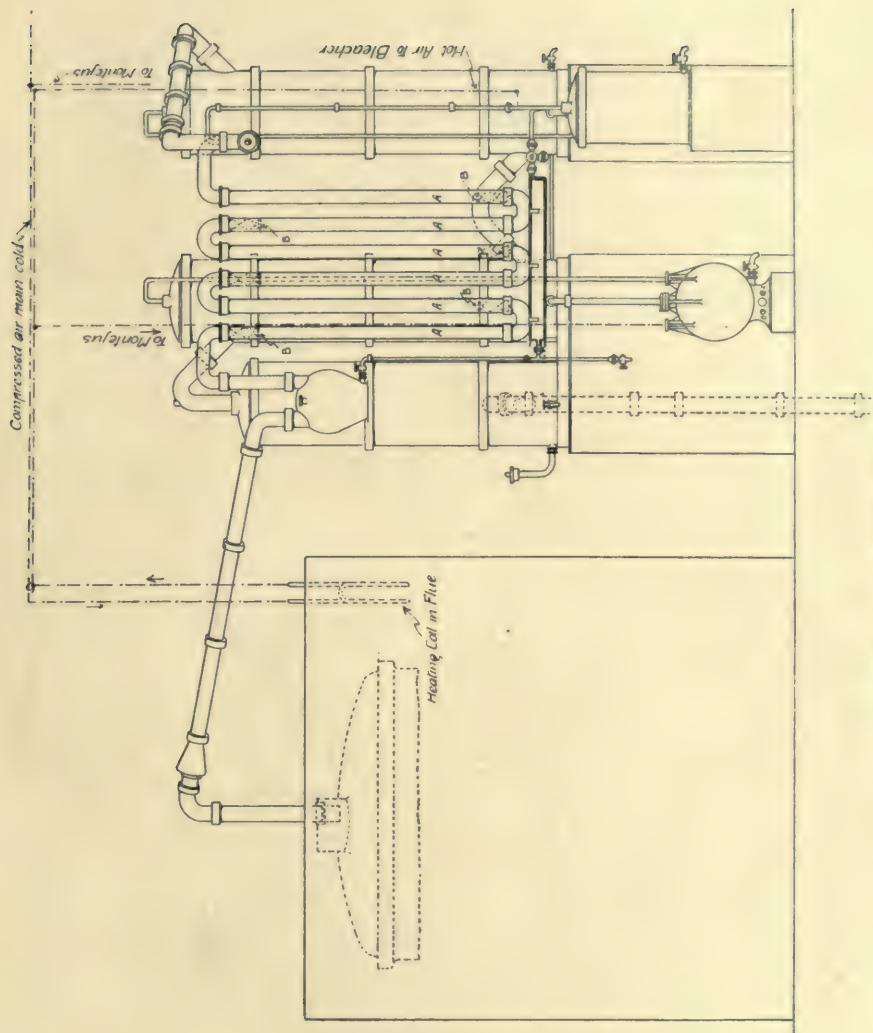


FIG. 168.—GUTTMANN PLANT.

air into the bottom of the bleacher, and the nitrous vapours that come away in their upward course have a good chance of being oxidised in the condensing system. In a discarded form of this type of condenser, small flanges were fitted to some of the "S" pipes, and the air (and at one time ozone) blown in direct, but this has since proved to be an unnecessary complication.

Silica Ware Condensers.—Figs. 165, 166 and 167 show the same class of reflux condenser illustrated in Fig. 164, but made in fused silica ware. These illustrations are supplied by The Thermal Syndicate, Ltd., of Wallsend-on-Tyne.

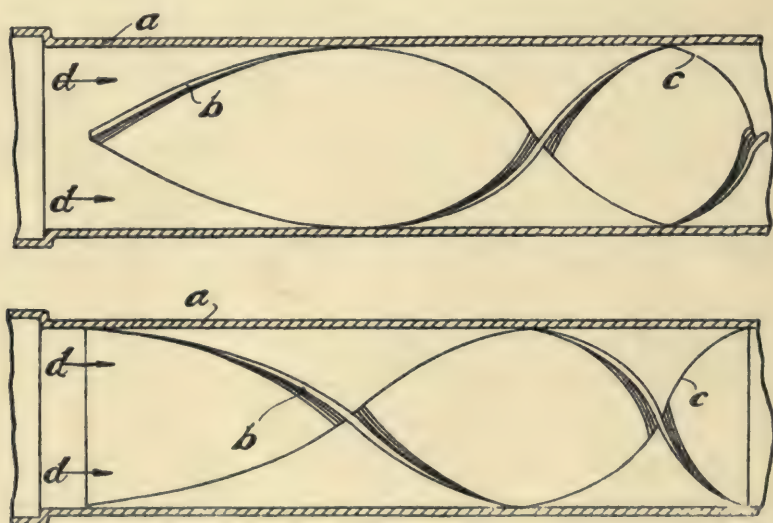


FIG. 169.

Figs. 166 and 167, it will be remembered, are taken from a paper read by Dr. Bottomley before the Newcastle Section of the Society of Chemical Industry on April 25th, 1917. The drawings are fairly self explanatory. It is interesting to note that the type shown in Fig. 166 is in great demand in Government factories and from it a high-class quality of nitric acid is obtained. The form shown in Fig. 166 is the ordinary 2-ton plant in which a charge can be distilled off in as short a time as eight hours. The weak point is the coil. This can, however, be easily repaired. The form preferred by many chemical engineers is the one shown in Figs. 165 and 167, where the reflux element "A" corresponding to the coil "B," Fig. 166,

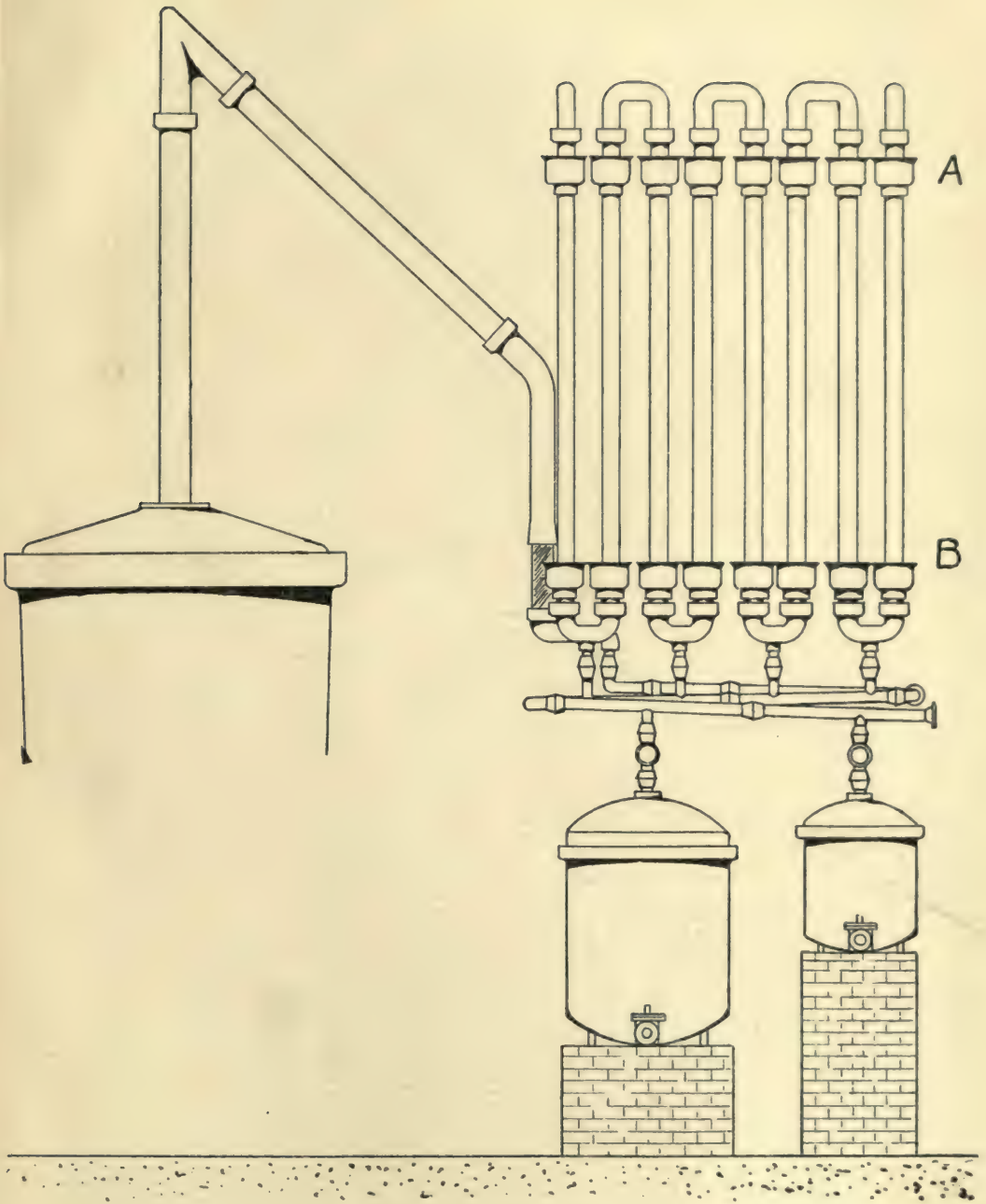


FIG. 170.—DURON PLANT.

is constructed of a series of "S" pipes as shown. Water cooling is, of course, applied to assist condensation in silica ware plants by fitting over the "S" pipe from the still a series of perforated water jets. In the case of the coils a canvas fabric is often hung either inside or outside, and kept wetted.

Guttman Plant.—Fig. 168 shows the well-known Guttman plant. In this plant the condensing pipes are placed vertically, which offers the advantage of allowing the condensed acid to flow rapidly from the surface

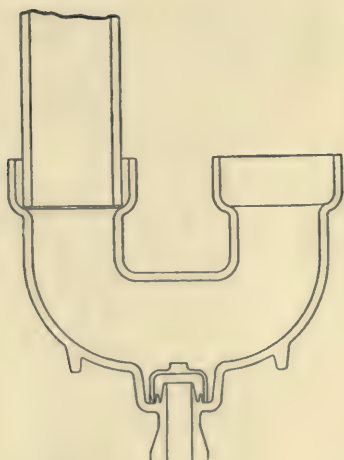


FIG. 171.

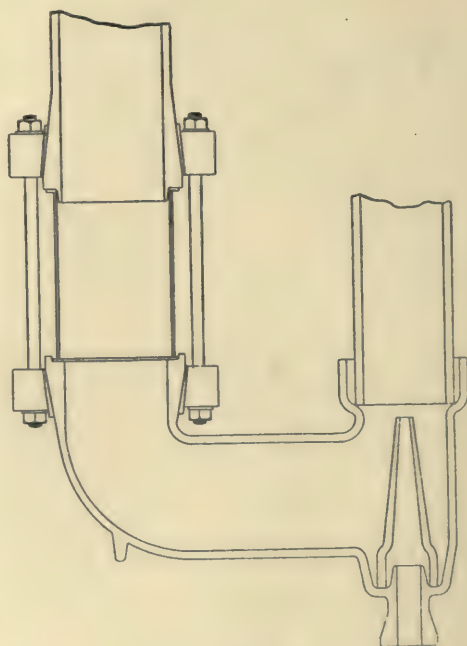


FIG. 172.

of the pipes away to the container, through liquid-sealed run-off pipes. A more efficient use of the cooling surface is thereby attained. In early forms of this plant the vertical condensing pipes were inserted in a water tank, but this method has now been discarded, and the plant is now supplied with each pipe fitted with its own water film sprayer at the top and a common catch tray at the bottom. This plant is the outcome of very many years experience, and in spite of many rivals it has maintained most of its original features. Some improvements have been made, such as the design of the collecting trough at the bottom. The design of the plant has been simplified and standardised,

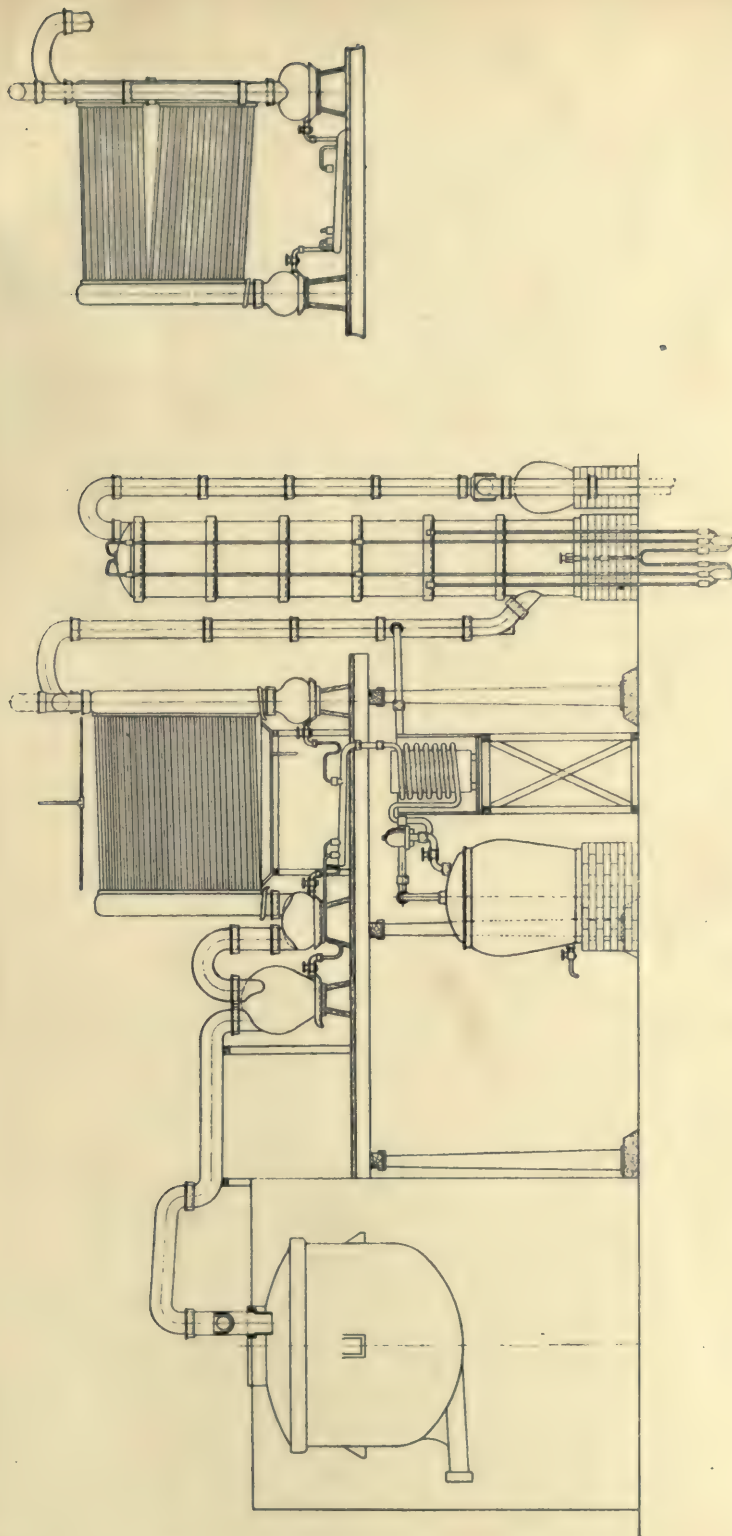


FIG. 173 — DOULTON NITRIC ACID PLANT.

and the intricacies of the earlier forms have been removed. It is interesting to note that the "Moore" gas producer is designed on similar lines, that is, that all condensing surfaces should be vertical to allow for the immediate separation of any condensed liquors. Objections to the vertical system of pipes has been urged on the ground that convection currents of hot air are formed which rise upward and surround the pipes, and thus prevent the cold air from gaining access to the cooling surface. There is some point in this in the case of ordinary air-cooled pipes, but with the film the objection vanishes.

The condenser pipes A may either be constructed in fused silica or in Ironac, preferably the latter, if a large output is desired. The efficiency of the cooling surface has been improved by the use of spirals B, Fig. 168 (British Patent No. 14165 of 1915). The theoretical increase in efficiency is about 30%, but in actual practice the improvement is more like 20%. A detail of this spiral is shown in Fig. 169.

Duron Plant in Ironac.—A similar plant known as the Duron system (and as made of "Ironac" by Messrs. Haughton's Patent Metallic Packing Co., Ltd.), is illustrated in Fig. 170. Its chief difference from the Guttman system lies in the method of withdrawing the condensed acid from the vertical pipes. Instead of using a single condensing trough the acid is tapped by a series of lutes into a delivery main, provision being made to separate the weak acid that is formed at the end of the operation. Fig. 171 shows the detail of the run-off lute in the vertical pipes in the Duron system. It will be seen the lute is simply inserted in a depression in the pipe enabling the acid to escape from the pipe without at the same time allowing any gas to flow that way. The cups "A" fitted at the upper end of the vertical pipes are all lead, and are pressed up on the iron alloy pipe. A series of perforations round the lower surface enables a film of cooled water to flow evenly down the pipe. Provided the area of outlet "B" is greater than the outlet area of "A" there is no danger of an overflow of water at the outlet.

Fig. 172 shows a detail of cone joints in Duron's system, and a very simple form of lantern which consists of a glass cylinder bolted between the end of two cone-ended pipes.

The bleaching of the acid in the vertical system of pipes is carried out by injecting a current of air into the bottom of the vertical pipe as shown in Fig. 172 or, as in the Guttman system, Fig. 168, it may be bleached continuously in the final bleacher container of the plant.

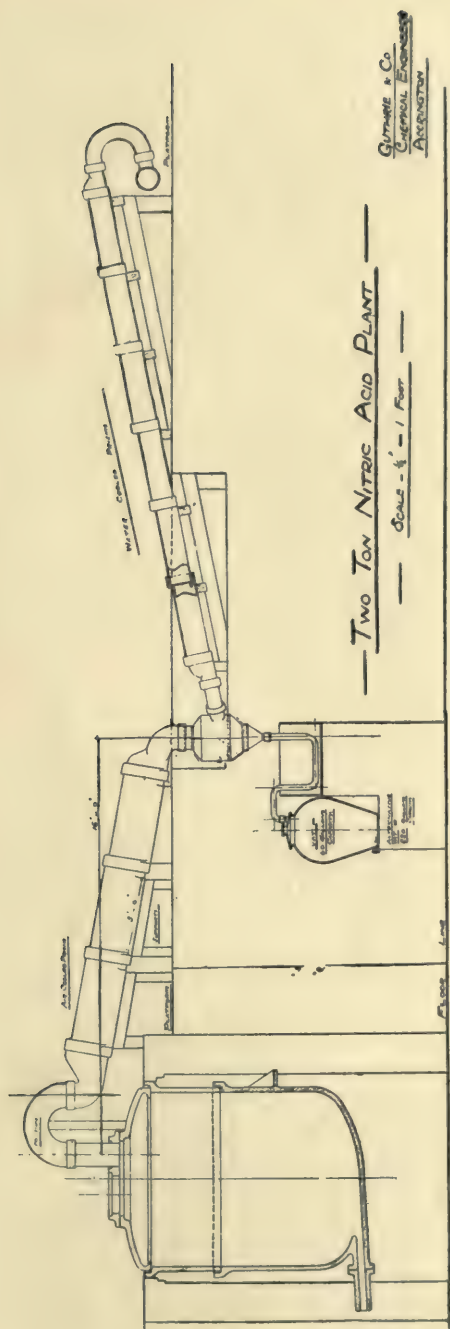


FIG. 174.—CERATHERM NITRIC ACID PLANT WITH PRISM CONDENSER, BY GUTHRIE AND CO.

The Valentiner system of condensation, which usually consists of an earthenware coil (or silica coil nowadays) and a series of Woulfe's bottles, is not in great use in this country. It is much in evidence in France. English manufacturers have not seen the advantage of instituting expensive vacuum pumps, and rely upon maintaining purity of product by careful management of the retort or separate bleaching. The form of Valentiner plant lately in use in a Northern works was designed so that the still could work under pressure with the condensing system under vacuum. This was done by inserting a throttle plate in the top of the still, and by its resistance enabled the withdrawing of the escaping gases to take place with consequent drop in pressure. There are, however, elements of danger due to the rise of pressure in the pot. It is to be noted that British Dyes, Ltd., have installed a large Valentiner vacuum plant at their Dalton Works, Huddersfield. It is said there is little difference between the efficiencies of this and the ordinary atmospheric process.

A further type of nitric acid plant is now being manufactured in this country, but has not yet been very widely adopted here. This is the Uebel system, and is chiefly in use in Germany and Italy. The process is carried on in three stages with three separate sets of condensers. The resulting acid is of good quality, but the plant is relatively very expensive, and requires high-class technical supervision.

The Hart condenser is still being fairly well used. The chief feature of the plant is a series of glass pipes fixed in a horizontal position in tubulures in two vertical stand pipes which can be either in earthenware, silicon iron alloy, or silica ware. It does not possess any specific advantages over the ordinary vertical or reflux type. It occupies small space, however, and the pipes being of glass, can be easily replaced, provided, of course, a suitable soft packing is used to make the joint. First cost is stated to be lower than with most of the other types. The Hart condenser and the standard 2-ton Hart plant as manufactured by Messrs. Doulton and Co., Lambeth. For works requiring small quantities of nitric acid Messrs. Doulton and Co. supply a standard plant in the form shown in Fig. 173.

"Ceratherm" Nitric Acid Plant. — Fig. 174 shows a "Ceratherm" nitric acid plant with patent cooling prisms, by Guthrie and Co., of Accrington, many installations of which are working satisfactorily. The makers claim distinct advantages for "Ceratherm" ware in nitric acid plant, and their claims appear to have been justified by experience under war conditions. Freedom from break-

age through thermal change, good condensation by reason of the relatively good thermal conductivity, lesser fragility as compared with silica ware, and lower weight as compared with ferro-silicon metal, are the principal points claimed for Ceratherm. In the plant illustrated in Fig. 174, Ceratherm water-cooled prisms (Fig. 175) are used, and it is claimed that these prisms offer great opportunities of rapid cooling, for example, in the case of the 5-inch prism the area of cooling is double that of the pipe of 5-inch bore. Prisms are also inserted where the effect of rapid cooling is most beneficial, namely, at the bottom of the reflux type of condenser. A small serpentine made either in Ceratherm or in a new intermediate body, which has properties in between stoneware and Ceratherm, and which is used where a less high quality material is desirable, completes the condensing apparatus. The prisms are very easy to spray with water, a rose on each side easily covering 90% of the surface of the prism. They can be made in such form as to obviate the use of coils for any class of work.

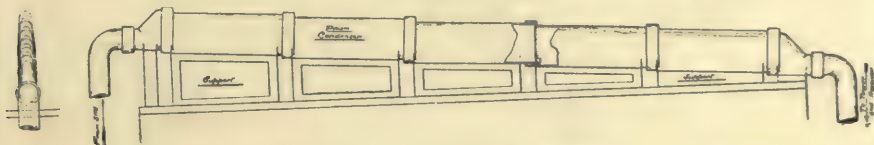


FIG. 175.—“ CERATHERM ” PRISM.

In the case of the Hart plant the use of Ceratherm headers (see Fig. 162) eases the whole plant, as in addition to being a safe means of conducting the gas so that there can be no fear of leakage or cracks of any kind, considerable emission of heat takes place, which is advantageous. The large double inlet pipes for the Hart plant are very satisfactorily manufactured in Ceratherm. Large quantities of the material have now been used on nitric acid plants for a considerable time, and the makers state that in not a single article has failure occurred through normal wear and tear of the plant.

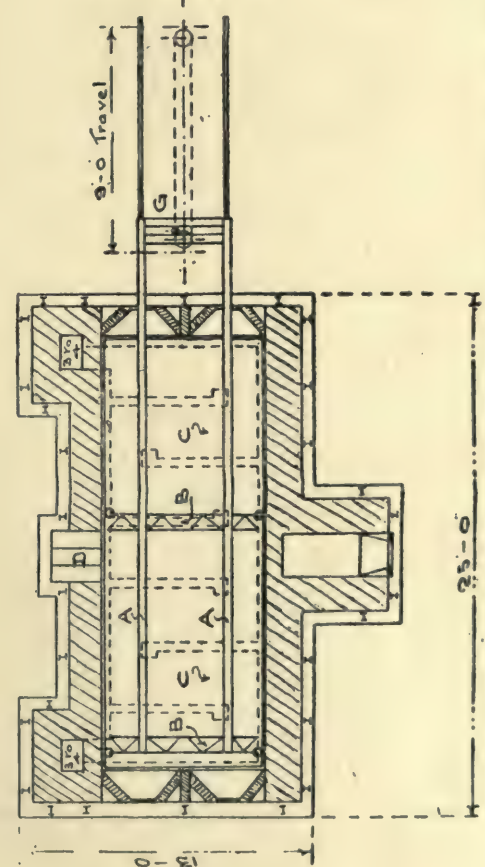
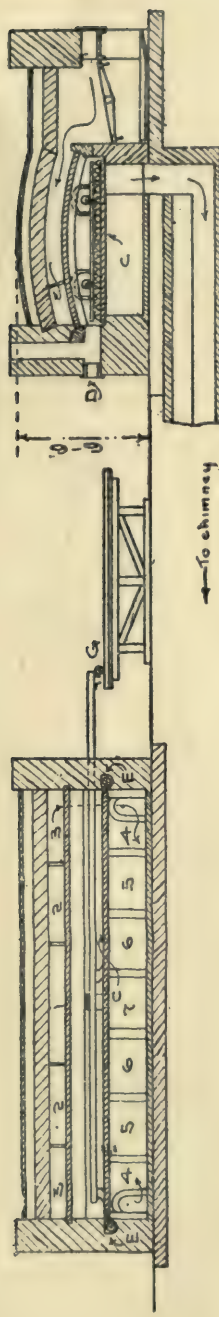
Joints.—Flanged joints have been discarded universally, the now standard form being a spigot socket joint, made with some suitable soft putty. The character of the putty mixture is a matter of some importance. It must, of course, be acid-resisting. It must not set so hard that a pipe cannot be removed without breaking, and it must be sufficiently hard to maintain the pressure of the gases inside. Soon after the outbreak of war, when many of the plants were being erected by firms with little or no chemical experience, and owing to the experience of the right method of making joints being kept in confined regions, attempts were made in Government factories to arrive at

some uniformity of practice. This generally has come down to the following:—Asbestos, small, 40 parts; asbestos fibre, 8 parts; whiting, by weight, 10 parts; a little tallow, and the balance boiled linseed oil.

Some firms substitute china clay for the whiting, which, on the whole, is preferable. Another common form is neutral silicate of soda (33% silicate), 19 parts of water, into which is mixed powdered asbestos until it forms a stiff dough, breaking clean in the hand. Barium sulphate may be added to produce a harder setting mixture, but must be used with care. For glassware and delicate earthenware this mixture may set too hard. For Tantiron or Ironac socket joints red lead and glycerine is recommended, made into a soft putty.

Packing.—For general purposes much difference of opinion exists as to the right kind of packing. Dr. Hurter some years ago did some classic work on the absorption of chlorine gas in towers which is recorded in the early numbers of the *Journal of the Society of Chemical Industry*. Of recent years the subject has been specially studied in the new atmospheric nitrogen fixation industry, where large volumes of air-diluted nitrous vapours require to be oxidised and absorbed to form strong solutions. Generally speaking, hard foundry coke was universally employed for gaseous absorption, excepting with nitric acid, in which case masses of broken earthenware, glass, and similar bodies were used. Closer study of the problem brought forward appliances based on the action of the fractionating column. Lunge's plate tower is a fair example. Later developments produced many forms of packing rings, balls, plates, etc. The best of these aims at exposing an adequate amount of wetted surface, together with as much free space for the gas to fill as possible. The free space should be disposed so as to form eddy currents and promote mixing and contact with the wetted surface. Greater attention is being given to these rules nowadays. Much of the earlier broken earthenware type of packing is being replaced by special cellular packing. Cells (see page 277) recently introduced by Guttman, are proving successful. Condensing systems constructed entirely of towers packed with these cells are usually 50% more efficient than when packed with bricks or rings. Many firms still follow the practice of mixing different fillings in the same tower. This is not recommended as a rule, though in the case of ordinary rings a thin layer of small broken earthenware helps to distribute better the absorbing liquid.

The Utilization of Nitre Cake.—Reference has been made to nitre cake as being one of the unsolved nuisances of the war. Owing to the scarcity of sulphuric acid the Government early used



- A Mechanical Rabbling Arrangement
- B Rollers of high speed tool steel (two sets) each of two rows of scrapers (Back row shown dotted.)
- C Air Iron Plates forming Hearth.
- D Special Charging Door for Salt and nitre cake mixture
- E Screw conveyors for removing Salt cake
- F Hydrochloric Acid Gas outlet
- G Mechanism - 2-4 H.P. required

NOTE:- The path of the furnace gases is indicated by numbering the compartments of the muffle and those under the hearth consecutively.

July 10th 1915

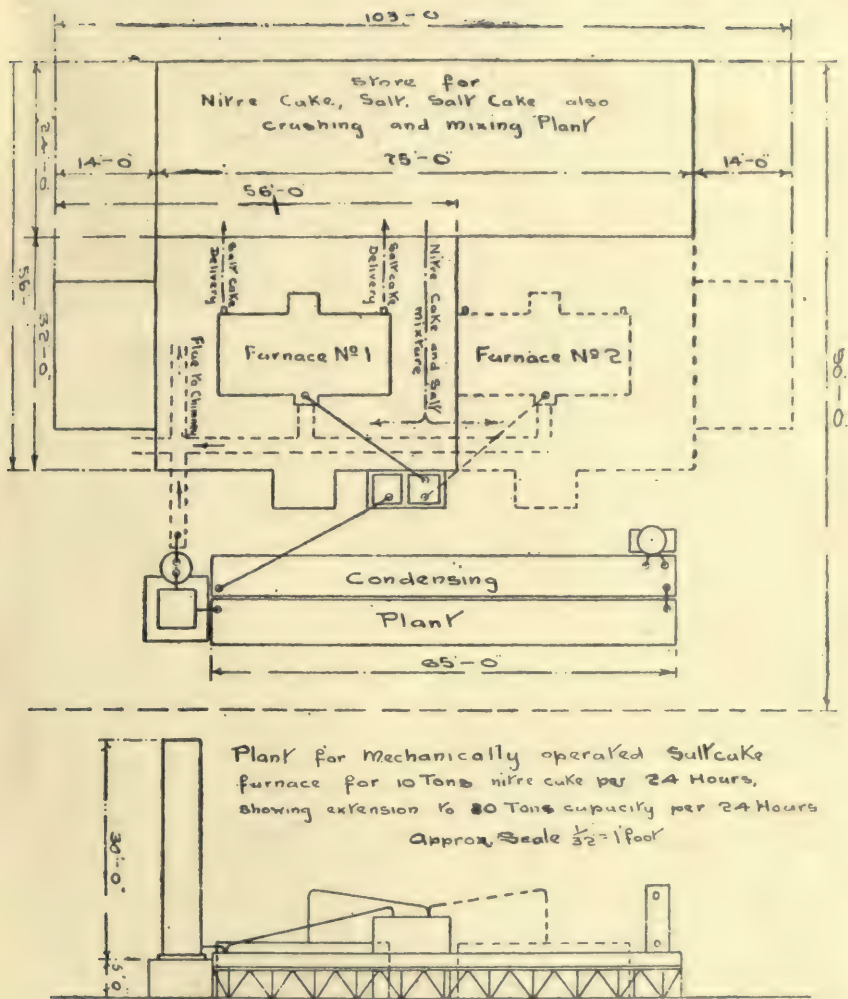
FIG. 176.—SIX MECHANICALLY OPERATED SALT-CAKE FURNACE FOR THE UTILIZATION OF NITRE CAKE.—TYPE A.

its pressure on manufacturers to make use of nitre cake as an acidifying agent in pickling, bleaching, grease refining, and in all processes where dilute acid was employed. Needless to say in ordinary times nitre cake would not have been considered. Nitre cake is essentially crude acid sodium sulphate. The pure acid sulphate contains 40·8% of sulphuric acid; the free acid found in nitre cake varies from 25 to 33%. Nitre cake contains also small quantities of free nitric acid, which from a pickling point of view are negligible. After many initial difficulties, some success has been made with brass pickling by nitre cake.*

In a discussion on the subject† at a meeting of the Nottingham section of the Society of Chemical Industry mention was made of many uses for nitre cake. A saturated solution was being used for pickling skins and refining grease. For the former purpose nitre cake did not behave like an equivalent quantity of sulphuric acid. When used for fat recovery in the wash water of wool it did not pay to recover the potash. This method of disposing of nitre cake is, after all, only a war measure, and represents no technical progress. The best practical scheme so far appears to be the use of nitre cake for the manufacture of hydrochloric acid, and has, of course, been in use for a number of years. Formerly nitre cake was charged in the ordinary salt-cake furnace together with the salt, no special plant being used at all. Recently careful attention has been given to the design of the furnace. An interesting, simple, and successful type of salt-cake furnace has been developed by Geo. Six (British Patent No. 4226, 1916), and is illustrated in Figs. 176, 177 and 178. Several of these furnaces are in operation designed to deal with 5 and 10 tons of nitre cake per day. The furnace consists of a simple form of muffle heated by an ordinary coke furnace which can be adapted for gas firing. The fire gases enter on top of the muffle at the centre, and divide into two streams, one zig-zagging to the right, the other to the left. At each end the two streams fall into a bottom flue divided by baffle walls, so that a staggered course is offered to the gases on their way to the centre, where they unite and go to the chimney. The salt is finely ground and thoroughly mixed with nitre cake broken to about $\frac{1}{4}$ in. size, and charged into the muffle, and drawn continuously along the bed by a mechanical rabble. The resulting salt cake is withdrawn at one or both ends by a screw conveyor. A reference to the satisfactory working of this

*J.S.C.I., Brownsdon on Nitre Cake for Pickling Annealed Brass, June 15th, 1917. p. 575.

†J.S.C.I., November 30th, 1917, p. 1216A.



NOTE - Open shed only required for furnace Building = 18 ft 6 inches.

FIG. 177.—SIX MECHANICALLY OPERATED SALT-CAKE FURNACE FOR THE UTILIZATION OF NITRE CAKE.—TYPE B.

furnace appears in the 53rd Annual Report on Alkali Works during 1916 (District V.—South Midland and Norfolk). Since this report was made the difficulties referred to have been overcome. The discussion on nitre cake mentioned above* probably also refers to this furnace. Dealing with the average composition of salt cake, Mr. G. E. Goober states that the average of 696 samples produced showed acidity as SO_3 1.65%; sodium chloride 1.81%. The control of the conversion is easy, and no trouble is experienced with the nitrous gases. The acid produced is about 35° Tw. (34.4%).

The Six Mechanical Salt-Cake Furnace.—By the courtesy of Mr. Georges Six the author is able to give the following particulars of the construction and working of his mechanically operated salt-cake furnace for the utilization of nitre cake. The furnace is made in two standard sizes, the output being 4 to 6 tons of salt-cake per 24 hours for the smaller size (type B, Fig. 177), and 6-10 tons of salt-cake per 24 hours for the larger size (type A, Fig. 176). The output varies with the quality of salt-cake required. The design of and the ground space required for a plant comprising two large furnaces (type A) will be apparent from the sketches. The furnace is of the closed or muffle type, and is continuous in operation. The bed consists of flat cast-iron plates on which the mixture is continuously rabbled by reciprocating rakes of special design. The mechanical arrangement is simple, and not liable to get out of order. It can be easily and quickly removed from the furnace for adjustment, and without taking it to pieces or dismantling any part of the furnace, and while the latter is still hot. The furnace can be used either for salt-cake manufacture in the ordinary way or for working up nitre cake with salt, without the use of added sulphuric acid. The quality of the resulting sodium sulphate and hydrochloric acid is quite satisfactory, and the fuel consumption is between 12% and 20% of the weight of bisulphate treated; gas firing is always an advantage. The working temperature is lower than that in hand-operated furnaces; 2 to 4 h.p. are required for each furnace. The salt-cake produced by the furnace is stated not to contain more than a total of 2% of free salt and sulphuric acid, and can be made purer. The fuel consumption will not exceed the equivalent of 4 cwts. of good coal per ton of sulphate. The strength of the hydrochloric acid produced will be between 35° and 36° Tw. reckoned at 15° C., and over 80% of the yield and upwards should contain less than 1% of

* *J.S.C.I.*, February 15th, 1913, p. 481.

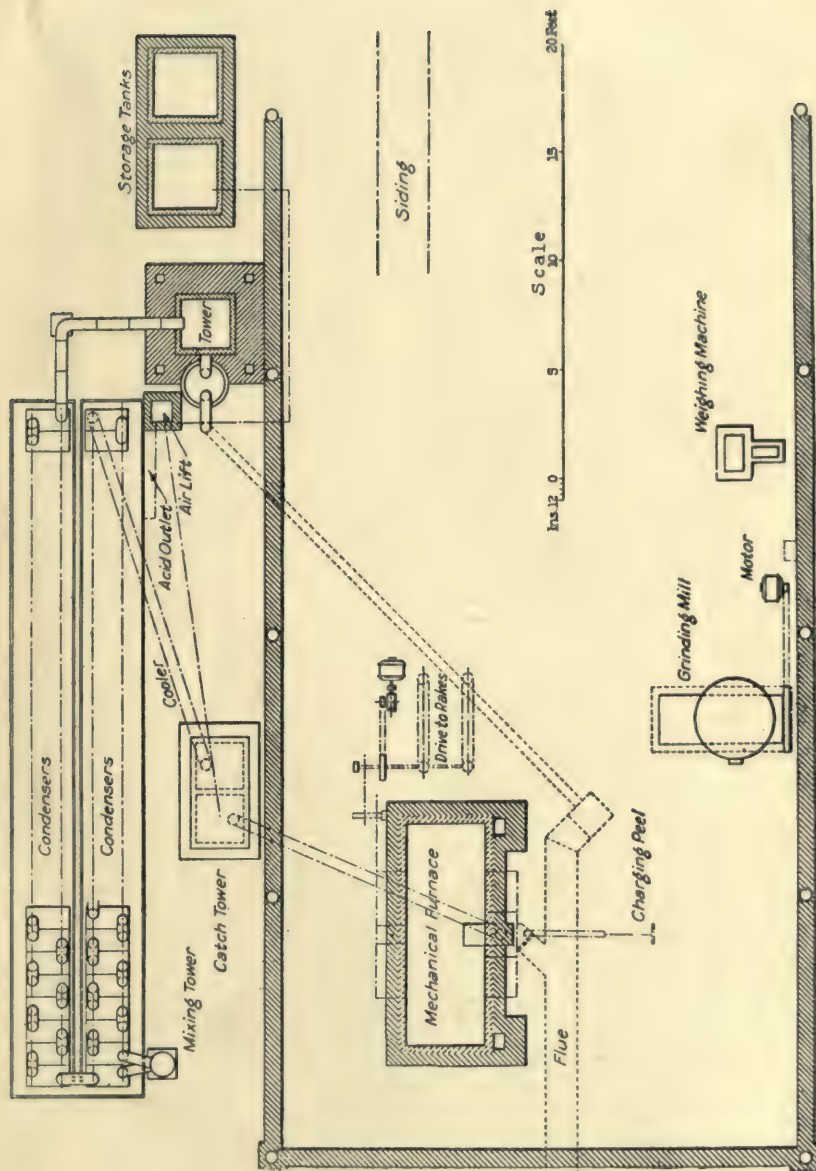


FIG. 178.

SO₃. An improved type of Cellarius condenser is used to attain this result. This condenser is made in two standard sizes having 18 and 25 sq. ft. respectively of water-cooled surface. The gas inlets and outlets being off centre, the length of path of the gases is increased, while the size, and therefore cost, of the connecting bends is materially reduced. The illustrations shows the approximate ground space required, but the plant can be arranged to suit an existing site or buildings. The following is an estimate of plant (June, 1918), comprising six mechanical salt-cake furnaces (type B), with condensing plant. Output: 96-120 tons per week of 28° Tw. hydrochloric acid, using nitre cake of about 28% free acid content. Six furnaces at, say, £650, £3,900; crushing plant, £800; condensing plant, £2,000; erection and contingencies, £1,700; total, £8,400. Extras: Chimney, £300; open shed for furnaces, £600; motors, water supply, etc., £600; storage plant for nitre cake, salt and salt-cake, pumping and small storage plant for acid, £1,200—£2,700; royalties, £675; plans and specifications, £225; total, £12,000. The following is an estimate of cost of production, in such a plant, of 28° Tw. hydrochloric acid, using mechanically operated salt-cake furnaces. In this estimate the prices of raw material have been liberally allowed for, while the value of the salt-cake to be sold is taken fairly low. The estimate should, therefore, be modified to suit local prices and conditions. The figures are based on an average output of 100 tons of 28° Tw. acid per week: Power, 7,500 K.W. hours at 1d. per unit, £31 5s.; process wages (6 men and 1 foreman per shift in 2 shifts), £33; general wages, £3 15s.; repairs, materials, £15; repairs, wages, £7 10s.; general expenses, supervision, light, water, etc., £11; depreciation, 15% on plant, £30; depreciation, 7½% on building, etc., £4; cost of operation per week, £135. Cost of Production.—50 tons of salt at 35/-, £87 10s.; 146 tons nitre cake of 28% at, say, 15/- per ton, £109 10s.; 10 tons of coke at 35/-, £17 10s.; cost of operation, £135; interest on capital, £20 10s.—£370 10s.; deduct revenue from 160 tons salt-cake, at 30/-, £240. Net cost of 100 tons of 28° Tw. acid, £130; cost per ton, £1 6s.; cost per carboy, say, 1s. 4d. The figures assumed in the above estimate are considered to be liberal, and would probably be improved upon in practice. As regards the cost of production, unit prices in the estimate must be modified to suit the conditions actually obtaining in each particular case. The plant is assumed to be in operation for 320 days in the year, and to use nitre cake containing 28% of free acid. If the plant is not intended to be an addition to an existing large works, but is to be self-contained, with its own

office buildings, works laboratory, and the like, it would be wise to allow for an additional £1,500 for various items under this heading and for excavations, roads, light railway tracks, weighing machinery and the like. The estimate includes the standard type of hydrochloric acid condensing plant by which acid of up to 35° Tw. is produced. This is usually preferable, as any surplus acid is more readily marketable, and a greater supply of acid can be stored in a given plant. If, however, the acid does not require to be of more than 28° Tw. strength, a small saving can be effected in the design of the condensing plant amounting to between £200 and £300.

In a paper by Mr. J. A. Wilson, Manager of the Devonshire Works of the Staveley Coal and Iron Co., Ltd., and Chairman of the Midland Section of the Coke Oven Managers' Association, the following reference was made to the Six furnace:—"Amongst other things the manufacture of which we have taken up since the commencement of the war are nitric acid and aniline. From the nitric acid plant we obtained as a by-product nitre cake or acid sodium sulphate—another of those materials which formerly were looked upon, in most cases, as fit only for the rubbish tip. . . Casting about for a use for this nitre cake, our chief decided that, as the aniline plant required hydrochloric acid and there was, owing to war conditions, an increasing scarcity of that article, he would instal a Six mechanical furnace. . . Several modifications have been made by us on the original design, partly introduced at the outset and partly suggested by experience obtained in the working of the plant. Briefly, it consists of a muffle furnace heated by coke oven gas, the hearth of which is formed of massive cast-iron plates, over which travel a set of mechanically operated rakes (Fig. 178). The mixture of nitre cake and salt, previously ground in an ordinary mortar mill, is charged by means of a scoop through an opening in the centre of one side of the furnace. The rakes seize hold of it, and gradually push it to the ends of the furnace, where the finished salt-cake falls into receptacles which are cleared out at intervals by means of an air-lift from the first four condensers, the idea being to wash out as much as possible of such impurities as sulphuric anhydride and chlorine. From the catch tower the acid fumes are passed through a main formed of stoneware pipes to a series of Cellarius condensers, a special form of condenser designed to expose a maximum surface of weak acid or water to absorb the fumes which pass over it in counter-current. At the end of this range of condensers is a tower 30 ft. high, built of Yorkshire stone, packed with Guttman cells in the lower part and with coke in the upper, in which the remaining

fumes are absorbed. The requisite pull on the gases is maintained by connecting up the outlet at the top of the absorbing tower to the chimney flue. The necessary amount of water to make acid of the required strength, which may be anything from 28° to 36° Tw., is passed down this tower and flows through the condensers in counter-current to the gas; the strong acid being drawn off at the fourth condenser from the gas inlet end. The acid is stored in tanks built up of Yorkshire acid-resisting stone slabs, the carboys in which it is sent out being filled in the wagons direct from the tanks by means of rubber piping. One advantage of a plant of this type over the ordinary form of salt-cake furnace, in which the heating gases are in contact with the materials to be heated, is that a stronger acid can be made than is possible with the latter type. With direct firing, acid of 28° Tw. is the maximum strength which seems to be obtainable, but on the Devonshire Works an acid of 35° to 36° Tw. is easily made."

Grossmann's Process for the Utilisation of Nitre Cake.—Dr. J. Grossmann has recently developed a process for the utilisation of nitre cake* in which this substance is baked in a muffle with nitre, with or without carbonaceous matter. Nitric acid or its equivalent corresponding to the free acid in the original nitre cake is evolved, which is partly condensed as nitric acid and partly absorbed as nitrite or nitrate. The following extract from Dr. Grossmann's paper gives the salient features of this process. He states that "Under certain conditions in a mixture of sodium nitrate, nitre cake, and charcoal or other suitable form of carbon, the sodium nitrate is completely decomposed with a steady evolution of nitrous fumes and without frothing, at a comparatively low temperature, and in the reactions which take place no reduction to nitrous oxide or nitrogen occurs, so that the yield of technically available nitrogen compounds is practically quantitative. The conditions under which this takes place are as follows:—The ingredients must be very finely ground and intimately mixed; the carbon must be present in excess, and a current of air must be passed through the system during the whole of the operation." Special treatment of the nitre cake is necessary to produce a friable and easily ground mass. The mixture was placed in a retort which was connected with a series of bottles containing a solution of caustic soda, and the mass was heated gradually to 250° C.

* *J.S.C.I.*, October 15th, 1917, Vol. xxxvi. 1035-37, and March 30th, 1918.

whilst a current of air was drawn through the system. . . . It was found that substantially all the nitrogen was recoverable in the caustic soda as nitrate or nitrite. . . . A typical test showed :

| | | | |
|-----------------------------|-----|-----|-------|
| Recovered as sodium nitrate | ... | ... | 54.5% |
| Recovered as sodium nitrite | ... | ... | 42.8% |
| Undecomposed | ... | ... | 0.9% |
| Loss by difference | ... | ... | 1.8% |
| | | | <hr/> |
| | | | 100.0 |

The residue produced is in a friable condition, practically free from chloride, and suitable for conversion to caustic in the Leblanc process. The coke used in the process is thus not lost. Similar treatment of nitre cake, but without the admixture of carbon, gave as a result :

| | | | |
|--|-----|-----|-------|
| As sodium nitrate | ... | ... | 85.2% |
| As nitrite calculated as sodium nitrate... | | | 13.1% |
| Undecomposed | ... | ... | 0.3% |
| Loss by difference | ... | ... | 1.4% |

The success of the process appears to depend chiefly on the ease of grinding nitre cake to a sufficient degree of fineness. Dr. Grossmann has found that at about 50° C. nitre cake becomes soft and can easily be crushed and ground. He had found it necessary in early experiments to add a small quantity of Na_2CO_3 to the melt of nitre cake to render the mass spongy, but in most cases this addition would not be required. The process has not yet been tried on the large scale, hence an opinion concerning its merits rests somewhat on conjecture as to after-war conditions. Captain Goodwin, in a written communication to the discussion on Dr. Grossmann's paper* has made a few comparisons between this process and the ordinary HCl process using the mechanical furnace previously described. In the first place Goodwin points out that the nitric acid process will not meet after-war conditions. Grossmann, on the other hand, in his reply to Captain Goodwin's criticism,† aims at stopping the waste of salt cake and sulphuric acid, since his process results in the formation of only one ton of salt cake as against three tons in the HCl process. On this point it should be remembered that the free acid in the nitre cake, whether used for making either nitric or hydrochloric acid, produces the same amount of

* *J.S.C.I.*, November 30th, 1917, Vol. xxxvi. p. 1161.

† *J.S.C.I.*, March 30th, 1918, Vol. xxxvii. pp. 103/4.

salt cake. The nitric acid originally made in the formation of the nitre cake stands to the credit of both processes. Grossmann's point is that for a given quantity of nitric acid produced his process forms less salt cake, and that it is the demand for nitric acid that determines the rest. The higher cost of condensing plant in the nitric acid process is set off against the more expensive mechanical furnace as compared with a simple muffle furnace working at 250° C. instead of 500° to 600° as in the HCl furnace. These various claims can of course only be substantiated in practice.

CHAPTER VI.

BENZOL AND TOLUOL RECOVERY AND TAR DISTILLATION PLANT.

The imperative call for vast quantities of benzol and toluol for the production of high explosives during the Great War dictated the lines of development of this class of plant, and the design of the recovery apparatus occupied the attention of chemical and gas engineers working under intense pressure to secure a vastly increased output in a minimum of time. The work thus done will necessarily determine the lines of future working, and in this chapter a summary of the general practice is given, followed by detailed descriptions of typical installations.

In a paper read by Mr. Geoffrey Weyman, M.Sc., before the North of England Gas Managers' Association on "The Effect of the War on Tar Works Practice," the processes introduced for the recovery of benzene, toluene and xylene are described, and from this description the following points are abstracted. One of the two processes aimed at the concentration of these bodies in the tar. The other involves the use of a wash-oil, and gives a more complete extraction. The former is at complete variance with the practice formerly. In a paper read before the Society of Chemical Industry in 1914, it was shown that when coal tar was separated from the gas at a high temperature, benzene was present in the purified gas to a far greater extent than if the tar and gas were allowed to cool together. Owing to the necessity of giving the gas a high intrinsic illuminating value, it was important to separate the gas and tar at as high a temperature as possible. Under the present process, the tar is cooled and then brought back into contact with the gas, which has also been cooled. In this way the tar becomes saturated at the tem-

perature of contact, and the lower this temperature is the greater is the concentration of the benzene and toluene in the tar. The distribution of the benzene and toluene between the gas and the tar is a definite one, depending on: (1) The original percentage of these hydrocarbons in the gas leaving the retorts; (2) the temperature of the last contact; and (3) to a certain extent on the composition of the tar. Complete saturation of all the tar per ton of coal with these hydrocarbons will not remove the whole of them from the gas, as the quantity of tar is far too small. Prolonged contact when once the tar is saturated is, of course, of no further use. The maximum efficiency of the process is about 50%, at which about 0.15 gallon of toluene and 0.9 gallon of benzene are left in the gas. All the way from the retorts to the condensers, the benzene and toluene are coming down from the gas with the other hydrocarbons which form the tar progressively as the temperature decreases; and if there happens to be such a long foul main that the tar and gas are already cooled when they are separated at the condensers, extra washing will not be of much benefit.

The following figures, showing the amount of benzene and toluene found in the tar drawn off in different stages of the cooling, illustrate these points:—

| By Volume. | Retort house main. | Water- cooled con- densers. | Air-cooled con- densers. | Before circula- tion. | After circula- tion. |
|--------------------------------------|--------------------------|-----------------------------------|--------------------------------|-----------------------------|----------------------------|
| Crude naphtha, 145% ... | 1.84 | 6.27 | 3.08 | 1.63 | 1.42 |
| Benzene, % | 0.4 | 3.5 | 2.0 | 0.9 | 0.3 |
| Toluene, % | 0.4 | 1.1 | 0.7 | 0.4 | 0.2 |
| Temperature of contact °C. | 120 | 15 | 15 | — | 115 |

The last two columns refer to the tar circulation carried out by pumping tar from the wells through the retort-house foul main.

The value of the process lies in the fact that in most cases the amount of benzene and toluene in the tar is more than doubled, and may be worked up with very little more trouble. The decrease in the illuminating power of the gas owing to the extraction is about 20%, and in calorific value about 3%.

The second process is much more efficient, giving up to 90% or more efficiency, but needs extra plant. It consists in washing the gas with an oil (usually creosote oil), which is pumped through scrubbers on the counter-current system in a similar manner to the ordinary

scrubbing for ammonia. The saturated oil is then passed through heaters to the crude still at a temperature of over 130°C . The lighter vapours pass off, and the crude products still remaining in the oil are blown off with live steam. After cooling, the oil returns to the scrubbers and is used again. The crude benzol so obtained varies in quality according to the amount of live steam used and the percentage of crude benzol in the benzolized oil. An ordinary quality contains about 10% of toluene, 48% of benzene, and up to 8% or more of naphthalene. Since the ensuing fractionation is not perfect, and some of the toluene is found in the benzol, and some benzene and xylene in the toluol, the actual result is a yield of about 55% of benzol, 9% of toluol, and 10% of solvent naphtha. The remaining 20% or so is left as still bottoms.

The crude benzol is worked into unwashed benzol, toluol, and solvent naphtha in a refining still provided with a good column and analyzer; the latter serving for a further separation of the vapours. The control of the "change over" points for these fractions is best kept by trial distillation in a standard Engler flask in the laboratory—samples of the distillates being taken from time to time from the stills. Temperature indications on the stills, though useful as a guide, are not sufficiently constant, owing to interference caused by small amounts of water vapour. When the trial sample on distillation gives 90% of its volume below 95°C ., a change is made to the toluol fraction, and this is continued till another trial sample gives 90% below 120° , when the solvent naphtha fraction is started. Distillation is stopped when 90% below 180°C . is reached. The still bottoms are run off and cooled to recover naphthalene. These fractions are then separately washed with acid, water, caustic soda, and then water again. The washed products are then re-distilled and more carefully fractionated, intermediate fractions being taken off between the benzol and toluol, and between the toluol and the solvent. Samples of products so obtained gave the figures shown in Table I.

Up to three gallons, and even more, of crude benzol per ton of coal may be extracted, although to do this the washing must be efficient, and something like 60 gallons or more of wash-oil per ton of coal must pass the scrubbers. The quality of the wash-oil is an important factor. Normal creosote has a composition like that shown in Table II. The large quantity of naphthalene is a serious drawback, and although the percentage may be materially reduced in course of time by the recovery of the crystallized naphthalene from the still bottoms, yet the

introduction of fresh oil to replace wastage will bring the percentage sufficiently high to increase the naphthalene content of the gas.

TABLE I.

Benzolized Oil—Crude Benzol to 145° C.—3.3 Per Cent.

| — | | | | Crude Benzol (with Column). | Benzol (Engler Flask). | Toluol (Engler Flask). | Solvent (Engler Flask). |
|----------------------|-----|-----|-----|-----------------------------------|------------------------------|------------------------------|-------------------------------|
| Drop point ° C. ... | ... | ... | ... | 45 | 79 | 109 | 135 |
| Below 80° C. ... | ... | ... | ... | 6.0 | 12 | — | — |
| 85 ... | ... | ... | ... | — | 96 | — | — |
| 90 ... | ... | ... | ... | 56 | 98 | — | — |
| 105 ... | ... | ... | ... | 69 | — | — | — |
| 117 ... | ... | ... | ... | 72 | — | 81 | — |
| 120 ... | ... | ... | ... | 74 | — | 94 | — |
| 140 ... | ... | ... | ... | — | — | — | 36 |
| 145 ... | ... | ... | ... | 78 | — | — | 89 |
| 150 ... | ... | ... | ... | — | — | — | 97 |
| Benzene, % ... | ... | ... | ... | 48 | 97 | 3.1 | <i>Nil</i> |
| Toluene „ ... | ... | ... | ... | 12 | 2.0 | 86.5 | about 1% |
| Xylene „ (about) ... | ... | ... | ... | 7 | — | 10.4 | 80,, |

To obtain relief from the naphthalene question, one may dilute the wash-oil either with specially prepared creosote oil or with blast-furnace oil (the latter contains little or no naphthalene, and will dissolve very much more than creosote oil); or one may let the naphthalene go forward in the gas and at the same time saturate the gas completely at normal temperature and pressure with some liquid such as paraffin.

TABLE II.

| — | | | | Wash Oil New. | Wash Oil in Use 21 Days. | Blast Furnace Oil. |
|----------------------|-----|-----|-----|------------------|--------------------------------|--------------------------|
| Naphthalene % ... | ... | ... | ... | 21.6 | 18.0 | trace |
| Specific gravity ... | ... | ... | ... | 1.037 | 1.050 | 0.957 |
| Below 200 ... | ... | ... | ... | 0.5 | 1.5 | 4 |
| 220 ... | ... | ... | ... | 16.5 | 4.5 | 7 |
| 240 ... | ... | ... | ... | 40.5 | 26.5 | 24 |
| 260 ... | ... | ... | ... | 61 | 51.5 | 40 |
| 280 ... | ... | ... | ... | 70 | 65.0 | 58 |
| 300 ... | ... | ... | ... | 78 | 72.0 | 72 |

The percentage of benzene and toluene in coal gas may in many cases be almost doubled by paying close attention to the retort-house. Roughly speaking, high heats and full retorts are the most favourable

conditions. Too low a heat will produce paraffins, which will spoil such benzol as is recovered; too high a heat or too much space above the charge will tend to convert the benzol into naphthalene, owing to the radiant heat from the retort surface, while the yield of ammonia may be seriously injured.

Mr. George Taylor, in a paper read before the Coke Oven Managers' Association, describes the plant necessary for oil washing. Tower scrubbers of about the same capacity as those used for ammonia extraction are usual, and oil is circulated through them in a similar manner to that in which liquor is circulated in ammonia scrubbers. There is, however, a growing tendency to adopt rotary scrubbers, usually of the brush type. These scrubbers are highly efficient, and by reason of the more intimate contact of oil with the gas a higher percentage absorption is obtained than is the case with tower scrubbers; also, the plant can be arranged so that the power expended in circulating the oil is less. Another advantage is economy of ground space. A rotary scrubber of 8ft. internal diameter, with eight to ten washing chambers, is capable of dealing with the gas from 200 tons of coal per diem. According to the quantity of coal carbonised, the circulating stock should be from 40 to 80 tons, and the fresh oil required to make good losses runs from 20 to 35 gallons per 100 gallons of benzol made—unless it is salty, when somewhat more will be used. It is generally admitted that for economy an exchange of heat should take place between the incoming rich oil and the hot debenzolised oil from the still. In some cases the rich oil is heated further by steam heaters. This is particularly the case where the still is being worked up to its full capacity, and space cannot be afforded to allow the dephlegmatory action of a colder oil. The stills themselves fall into two classes: one in which powerful steam heaters are placed in a chamber at the base of the still, with the intention of drawing the effluent oil, and the other which works with wet steam only. In the latter type provision should be made for separating the water from the oil whilst still hot. Vapours of water and benzol leave the still at about 100° to 112° C., and must be partially condensed or dephlegmated in order that a 65% benzol may be obtained. The fraction which condenses at a temperature of less than 95° C. is the required material. The heavier matters consist of water, naphthalene, light oil, and phenols, and are usually small in quantity. In connection with the condensing of the benzol it is highly important that no back pressure should be thrown in this process. If this is not the case, difficulty will be experienced in denuding the oil of benzol, and losses will take place. Table III gives working

data obtained from plants in various parts of the country. It can be seen from these figures that a high saturation of oil, where consistent with a good return gas, is much to be desired; also the oil should certainly be heated to 110° C. in the still. Should this not be the case, and the effluent oil contains over 0.5% of benzol, the steam supply should be superheated. From information to hand concerning two plants, the introduction of superheated steam increased the yields of benzol about 5%.

TABLE III.

| | A. | B. | C. | D. | E. | F. | G. |
|---|----------------|---------|--------------|--------------|--------------|-------|---------------|
| Benzol in 10,000 cb.ft. unscrubbed gas | 2.5 to 2.7 gls | 2.5 | 2.32 | 3.0 | 3.3 | 2.52 | 3.0 |
| Benzol in return gas | 0.4 to 0.75 „ | 0.5 | 0.07 | 0.09 to 0.15 | 0.05 to 0.15 | 0.60 | 0.25 to 0.40 |
| Benzol in rich oil | 3.6 % | 2.5 % | 4.5 % | 4.5 % | 3.5 % | 3.1 % | 2.75 % |
| Benzol in debenzo- lised oil from still | 0.6 % | 0.3 % | 0.2 to 0.3 % | 0.8 to 1.2 % | 0.5 % | 0.9 % | 0.25 to 0.9 % |
| Temperature of oil passing to still ... | ? | ? | 108° C. | 120° C. | 85° C. | ? | 15.25° C. |
| Temperature of oil issuing from still | 110° C. | 105° C. | 112° C. | 95° C. | 105° C. | ? | 104° C. |

Basic impurities are easily removed by acid washing, but if it is necessary to remove thiophene also, the acid washing must be repeated and prolonged until the laboratory sample gives a negative result by the isatin test. In any case, the completion of the washing should be ascertained by distilling a sample from the washer, and treating the distillate corresponding to 90% benzol with 10% of pure sulphuric acid. The acid washing is followed by a water washing. Here care must be taken to avoid a violent agitation, but the water should rather be used to rinse the sides of the washer, as some benzols have a tendency to emulsify with water, and cause large washing loss. The acid washings treated with water separate into solid resinous matter and an acid liquor, from which pyridine may be separated by treatment with ammonia liquor, and the resulting sulphate solution used in the sulphate house. The benzol is then washed with an excess of 20° Tw. caustic soda solution, which removes the traces of sulphuric and sulphurous acids and any phenols that are present. The caustic solution may be used until spent.

The still employed for "blowing over" the benzol is of 3,000 to 4,000 gallons capacity, has twelve to fifteen trays in the fractionating column, and is provided with direct and indirect steam heating. The charge may be considered as worked off when the distillate boils at

200° C., or when naphthalene becomes evident. The once-run benzol must then be separated into commercial fractions by distillation in a rectifying still. Many types of these have been erected, the following being the essential features:—The body of the still should be of the short horizontal, cylindrical shape, and heat supplied by a large number of steam pipes running through the still and a wet steam coil. The fractionating column should be of ample capacity, the two main varieties now at work being the cap and plate introduced into benzol distillation by Heckmann, and the perforated plate originally adopted in Coffey's still. Differences in detail are found in the size and shape of the caps, depth of seal, position and shape of overflow; also in the number and size of holes in the perforated plate. It is worth noting that whilst newcomers in this field erect complicated plant, with cap-and-plate rectifying columns, benzol distillers of old standing show a preference for simple plant and perforated plate column. As a number of stills of the latter kind are successfully producing pure benzine and toluene, it would appear that the opinion prevailing in some quarters that only the cap-and-plate column will produce a pure product is scarcely correct.

The chief products obtained by distillation of benzol are shown in Table IV.

TABLE IV.

| Commercial name. | Per cent. of pure | | | | Specific gravity 15½° C. | Range of distillation at barometric pressure 760 mm. |
|-----------------------------|-------------------|----------|---------|---------|-----------------------------|---|
| | Benzene. | Toluene. | Xylene. | Cumene. | | |
| 90 per cent. benzol | 84 | 13 | 3 | — | 0.880 | 90 p.c. between 81 and 100° C. |
| 50 per cent. benzol | 43 | 46 | 11 | — | 0.875 | 90 p.c. between 89 and 120°; 50 p.c. at 100° C. |
| Commercial toluol | 15 | 75 | 10 | — | 0.870 | 90 p.c. between 107° and 120° C. |
| Solvent naphtha | — | 5 | 70 | 25 | 0.875 | 90 p.c. between 135 and 160° C. |
| Heavy solvent naphtha ... | — | — | 5 | 80 | 0.920 | 90 p.c. at 190° C. |
| Benzol for pure benzene ... | 95 | 5 | — | — | — | 90 p.c. at 90° C. |

The points at which the distillates should be changed into different receivers may be ascertained in two ways. The approximate quantities of each distillate are usually known. If, therefore, the tank into which the distillate is running gives the correct distillation test the receiver should be changed. This necessitates the constant attendance of someone able to make and interpret the tests. Alter-

natively, a litre sample may be fractionated in the laboratory, and the results being compared with the work results, it is possible to forecast the yields from future distillations with a fair degree of accuracy.

The plant used for toluene extraction at the Bradford Road Works of the Manchester Corporation is described as follows by Mr. H. C. Applebee in a paper read before the Manchester and District Junior Gas Association. "After leaving the purifiers, the gas is led through tower scrubbers, each of which is 55 ft. high and 7 ft. 6 in. diameter, and of which there are six. These are in three pairs, so arranged that they can be worked in two sets of three each, each of the three being arranged with bye-passes which will cut out any or all of them. Commonly the scrubbers are worked in two sets of three each, the stream of gas being divided. The wash-oil enters the system at the top of the last pair, finds its way to the seal-pots at the bottom, and thence overflows to the seal-pots of the second pair. From these it is circulated (by pumping) through the second pair; the overflow passing on to the first pair of seal-pots, from which it is circulated through the first pair of towers, where the gas enters. The oil overflows from the first pair of seal-pots and runs to the rich oil well. Thus gas and oil travel in opposite directions; the fresh oil being the last to meet the gas. It is evident that many conditions will affect the amount of products recovered from the gas. Those principally concerned will be: (1) The amount of wash-oil used; (2) the quality of the oil; (3) the temperature of the oil and of the gas; (4) the circulation of the oil; (5) the 'mechanical' efficiency of the scrubbers (including such items as their packing, number in use, etc.). There is evidently a practical limit to the amount of wash-oil which can be used; and the cost of dealing with excessive quantities of rich oil would outweigh the possible slight advantage which might be gained. At Bradford Road the amount of wash-oil used has varied from 70 to 105 gallons per ton of coal carbonized; and it is usually considered that from 60 to 70 gallons are necessary for efficient scrubbing. The best results have been obtained by using 105 gallons, and reductions of the amount of wash-oil to amounts below 70 gallons to the ton have invariably led to reductions in the amount of crude benzol recovered. The oil in use suffers deterioration by reason of the fact that, in the subsequent recovery of the crude benzol, part of the creosote is lost (amounting to about 20% of the crude benzol recovered). As a consequence, the specific gravity of the oil gradually rises, and the distilling points become higher, until a point is reached when it becomes advisable to change

the oil. It is considered advisable to do so when the specific gravity reaches 1.06 to 1.065, or the distillation shows less than 50% at 300° C. Just as in the 'C' process temperature is an all-important factor, so here it is advisable to keep the temperature of both oil and gas as low as possible. Any appreciable rise in temperature in the system results in a corresponding decrease in crude benzol production. Normally the oil entering the scrubbers is at about 25° C., at which temperature it is safe to say there is no serious loss. It is generally found that a suitable degree of saturation of the wash-oil is reached when the oil contains about 3% distilling up to 120° C. This point is to some degree regulated by the amount of circulation in the last two pairs of scrubbers, and, of course, by the amount of fresh wash-oil used. One would anticipate that, other things being equal, the larger the circulation the better the efficiency and the higher the degree of saturation. On the other hand, too great a degree of saturation may cause loss owing to the 'mechanical' vaporization of the lighter constituents by the passage of the gas through the oil. Our practice is to pass four or five times the amount of oil down the first and second pair of scrubbers than passes down the last. As to the packing of the towers, I have little to say. The arrangement will aim, obviously, as in any scrubber, at presenting the largest possible surface to exposure. We have consistently obtained better results by dividing the stream of gas through the two sets of three scrubbers than by passing all the gas through a single set. This is to be expected, since double the surface is available for absorption. It is evident that occasional tests should be made to ascertain the efficiency of the scrubbing. There are in common use for this purpose the following methods. First, a convenient quantity of gas is passed through a series—say, three or four—of wash bottles containing creosote or oleic acid, the naphthas being absorbed. The quantity of the latter is then determined by distillation. In the case of creosote, the oil should first be distilled to 200° C., while the oil from the test is distilled up to 200° C. or, alternatively, a 'blank' test may be made with the same creosote, the result being deducted from that obtained in the test proper. In the case of oleic acid, the naphthas are steam distilled from the 'enriched' oil. Oleic acid not being volatile in steam, the resulting naphthas directly represent the amount contained in the gas. We have also successfully applied Dr. Coward's method, which, however, requires special (and costly) apparatus. The method consists essentially in the introduction into a vacuum vessel of a quantity of the gas under examination at atmospheric pressure and temperature. The condensible

products (consisting of the naphthas) are then frozen out by a mixture of solid CO_2 and ether, the uncondensed gases being pumped away by means of a mercury vacuum pump. The vessel is then warmed up to atmospheric temperature, and the pressure noted. The percentage of condensible products is proportioned to the pressure. The amount of naphtha contained in the gases at the inlet and outlet of the scrubbers having been estimated by one or other of these methods, the efficiency of the scrubbers is readily calculated. Our tests have given the following results:—

| | | | | | | | % |
|--|---|---|-----|---|---|---|-----------------------|
| With three scrubbers in use and 74 gallons of wash-oil per ton ... | | | | | | | 75 to 78 |
| „ three | „ | „ | 108 | „ | „ | „ | 82 to 84 |
| „ six | „ | „ | 108 | „ | „ | „ | up to 88% efficiency. |

The decrease in candle power has varied from five to eight candles, according to the wash-oil used. The decrease in calorific value has been less marked, varying from 5 to 7%, according to the conditions in force at the time. So far I have spoken only of coal gas, but recently a new departure has been made, and we are now scrubbing carburetted water gas. The coal gas is taken through one set of three scrubbers; the other set being utilized for the carburetted water gas. The whole work is conducted on exactly similar lines to those for coal gas, and the general remarks already made apply equally well."

Stills.—The stills used at Bradford Road, and described by Mr. Applebee in the paper quoted, are of the continuous type, erected by Messrs. Simon-Carves, and consist of six parts—the still proper, the heat interchanger, the dephlegmator, the superheater, and two tubular condensers. The rich oil is fed to the still by pumps, and passes, first, to the first condenser, where it helps to cool the vapours leaving the still, and is itself warmed to about 50°C. ; second, to the dephlegmator, where it reaches 65°C. ; third, to the heat interchanger, where the temperature rises to about 80°C. ; fourth, to the superheater, where it is steam heated by closed steam to about 130°C. ; fifth, to the still proper, where it is brought into contact with the open steam; and, lastly, through the heat interchanger, and so away to the oil-coolers, which consist of gilled pipes water-cooled. The vapours leaving the still proper pass through the dephlegmator, leaving the latter at about 95° to 100°C. , passing on to the first condenser (which is cooled by the incoming oil), and finally to the last condenser, which is water-cooled,

and where the vapours are finally condensed. The feed pumps are regulated to work at a set speed, the quality of the crude benzol produced being largely determined by the amount of open steam introduced into the still. This is regulated to give a definitely determined temperature to the vapours between the two condensers. Once this temperature is decided upon and the conditions set, the still works quite regularly, and requires comparatively little attention. It is customary to produce benzol testing 65% at 120° C. when distilled from a retort with the thermometer in the liquid; and this practice is followed at Bradford Road. The crude benzol is next partially fractionated. For this purpose a Dempster still is used, having a capacity of about 3,000 gallons. Crude benzol testing 65% at 120° C., as above, yields, roughly, 60% of once-run benzol and 15% of once-run toluol, these amounts varying more or less according to the method of fractionation. The residues (or the 'bottoms' as they are termed) which are left in the still after the desired fractions have been distilled off, are run into shallow trays, where, on cooling, the naphthalene separates out. The oil which is drained away from the naphthalene, or 'creosote salts,' is returned to the wash-oil, the naphthalene being a marketable article. The once-run benzol and once-run toluol are then 'washed' separately. The washer employed is of the usual cylindrical type, tapering to a cone at the bottom. Its working capacity is about 2,500 gallons, and it is fitted with a belt-driven mechanical agitator. The acid used is sulphuric (concentrated oil of vitriol), its strength being as near 1.84 sp. gr. as possible, and is best added in two portions. The practice is as follows: Charge to the washer, 2,500 gallons. The first acid (five gallons) is agitated for fifteen minutes. This has the effect of removing traces of water, and some of the more basic impurities. After settling for a short time, the acid is drawn off, and the bulk acid (46 gallons) added. This is agitated for forty minutes, and allowed to settle for thirty minutes. The second acid being drawn off, water is run through the charge without agitation to remove the bulk of the remaining acid. This operation is repeated, after which the washer is agitated for ten to fifteen minutes with a further quantity of water—a second agitation with water is given if necessary. Caustic soda solution of about 20° Twaddell is next agitated with the naphtha for from fifteen to twenty minutes. After standing for about thirty minutes, the soda is run off, and the bulk finally agitated with water for a short time. The loss on washing is estimated at 7%. This, of course, will vary with different qualities of crude benzol, as will also the amount of acid required to produce a given degree of refinement. Some coal gas will yield a crude

benzol containing large quantities of impurities. Consequently, large amounts of acid are necessary, and the loss in washing will be correspondingly high. It was thought likely that the introduction of crude benzol produced from carburetted water gas would increase the difficulties in washing the once-run products. These fears, however, have not so far been realized, and the increased loss is comparatively small. After the washing process, naphtha is run into the final still, where the process of fractionation is carried further. The still is again of the Dempster type, with a capacity of about 3,000 gallons. The practice here will vary somewhat, according to the quality of the naphtha to be treated, the type of still used, and the products desired. Commercial toluol should contain 75% of toluene (tested by Dr. Colman's method), and no difficulty has been experienced at Bradford Road in meeting this requirement. In all processes of final fractionation there are more or less intermediate fractions, which are put back for re-working and, as in the preliminary fraction, when all the desired products have been distilled off, the ultimate residues are run to the naphthalene trays, where the naphthalene is again separated.

The Intermittent Still.—The recovery and fractionation of benzol from coal gas is described at considerable length in a paper by Mr. W. Diamond read before the North of England Gas Managers' Association, from which the following extracts relating to plant are taken.

The intermittent still is a large cylindrical tank, having a capacity of 30 to 40 tons, with a dome top and vapour outlet. The vapour outlet is fitted with a cast-iron swan neck, which leads to a condenser and receivers. Inside the still there is fitted a 2-inch wrought-iron coil the full height and circumference of the still, which is coupled to a steam-main, and at the bottom to a steam-trap. There is also lying across the bottom inside a single pipe, which is closed at one end, and coupled to the steam-main at the other. In the part lying across the bottom there are innumerable $\frac{1}{4}$ inch perforations. The usual sludge, etc., valves are fitted. The only drawback to this kind of plant is the large ground space required, and the considerable larger quantity of wash-oil in storage. They have distinct advantages over the continuous process in some ways, but the continuous plant is the one in general use at the present time.

The Continuous Still.—Continuous stills are of varied design ; but, broadly speaking, they consist of preheater, heater proper, and still coupled to the condenser and receivers. The heaters may be chambers

fitted with internal steam coils to raise the wash-oil to the required temperature. The preheaters are also condensers, in so far as the cold oil from the scrubbers is heated by the debenzolized oil from the still and *vice versa*. The still should consist of a bottom chamber and a vapour outlet on the top, coupled to a series of circulating trays, fitted one on the other, of about 3 feet diameter and 12 inches in depth, with a luted seal inside, over which is placed a toothed cap or cover. The surplus condensate in each tray is carried to the next lower tray by means of a short pipe, and eventually is conducted into the still. Inside, and along the bottom of the still, is fitted an open steam-pipe, as in the intermittent type. The number of trays in the column is decided by the requirements of the refiner. The top tray is fitted with a 4-inch outlet pipe, which is coupled to a condenser. The condenser may be of any preferred type, but must be absolutely tight. The outlet pipe from the condenser is coupled to a separator, in which are separated the water and benzol which have been vaporized in the still and condensed in the condenser. These separate by their own gravity, and are conducted to where they are required.

The continuous process for the production of crude benzol is as follows. The new wash-oil is pumped out of its store-tank into the base of the last scrubber, and is from there pumped by another pump to the top of the same scrubber, and counterflow to the gas. As the wash-oil is being continually pumped into the scrubber the same rate of flow of benzolized oil goes over the seal into the base of the next scrubber, and, after circulating there, gravitates to a tank which is termed the benzolized oil-tank. A third pump now draws it through a pipe through a superheater. This is formed by two pipes—a smaller inside a larger one. Through the inner pipe is drawn the benzolized oil, and surrounding it flows the debenzolized oil from the still. After this interchange of heat, the benzolized oil is drawn through the heater proper, where it is heated to about 130° to 140° C., and then forwarded into the still. The vapours and steam ascend the column, and are fractionated somewhat by the baffling in the trays by the bonnets. The mechanically carried stuff, of course, is caught by the inspringing of the vapours in the trays on the bonnets, and there, together with the higher boiling benzols, gravitates through the seal pipes down the column into the still. The lighter vapours find their way into the condenser, are condensed, and find their way into the separator, and so forward to the store-tank.

From the benzol outlet a sample is taken and tested in the laboratory. If it should be found to be above 65%, or any desired

strength, a little open steam is admitted in the still, which helps to liberate the higher boiling homologues, and weakens or lowers the percentage quality of the distillate. Should the benzol not be of sufficient strength or quality, the wash-oil is circulated through the scrubbers for a longer period, and afterwards only heated to a much lower temperature, and no open steam admitted, so as to liberate the lower-boiling products. From time to time samples are taken and tested, and the plant regulated according to the result of the test. A distillation test of crude benzol is as under :

| | | | | |
|---------------------|-----|-----|-----|-------------------------|
| Specific gravity... | ... | ... | ... | .898 |
| At 110° C. | ... | ... | ... | 37 per cent. distillate |
| 120° C. | ... | ... | ... | 65 " " |
| 160° C. | ... | ... | ... | 78 " " |
| 190° C. | ... | ... | ... | 85 " " |

This is the product from which is obtained all the refined products of benzene, etc. But it is quite impossible to obtain them by a single fractionation; the crude benzol must be refined as much as possible. To do this it is run into a pot-still, fitted with a column in the vapour outlet similar to the wash-oil still; but inside this still is fitted a steam-coil, also an open steam perforated pipe. There is also the requisite condenser, receiver, and separator. Steam is put into the coil, and the intermediate benzol is heated; the vapours passing through the column as previously described, condensed, etc. This coil is kept hot as long as any distillate comes over. The residue is tested when this ceases, and tested to 190° C. If there is any distillate under this temperature, open steam is admitted and carried on until by test the residue is shown to contain no distillate under 190° C. The residue is now run into cooling tanks, and when cold disposed of as creosote salts or any convenient requirement. By this distillation the product will have become considerably refined, as is shown by the following test :

| | | | | |
|------------|-----|-----|-----|-------------------------|
| At 100° C. | ... | ... | ... | 63 per cent. distillate |
| 110° C. | ... | ... | ... | 74 " " |
| 120° C. | ... | ... | ... | 80 " " |
| 160° C. | ... | ... | ... | 89 " " |

Fig. 179 shows the benzol and naphthalene recovery plant installed in 1916 at the Etruria Gas Works, which is described by Mr. Harold E. Copp in a paper read before the Midland Association of Gas Engineers and Managers. In describing the working of this plant, Mr. Copp observes that "One of the first difficulties encountered was due to the corrosion of the inner steam chambers

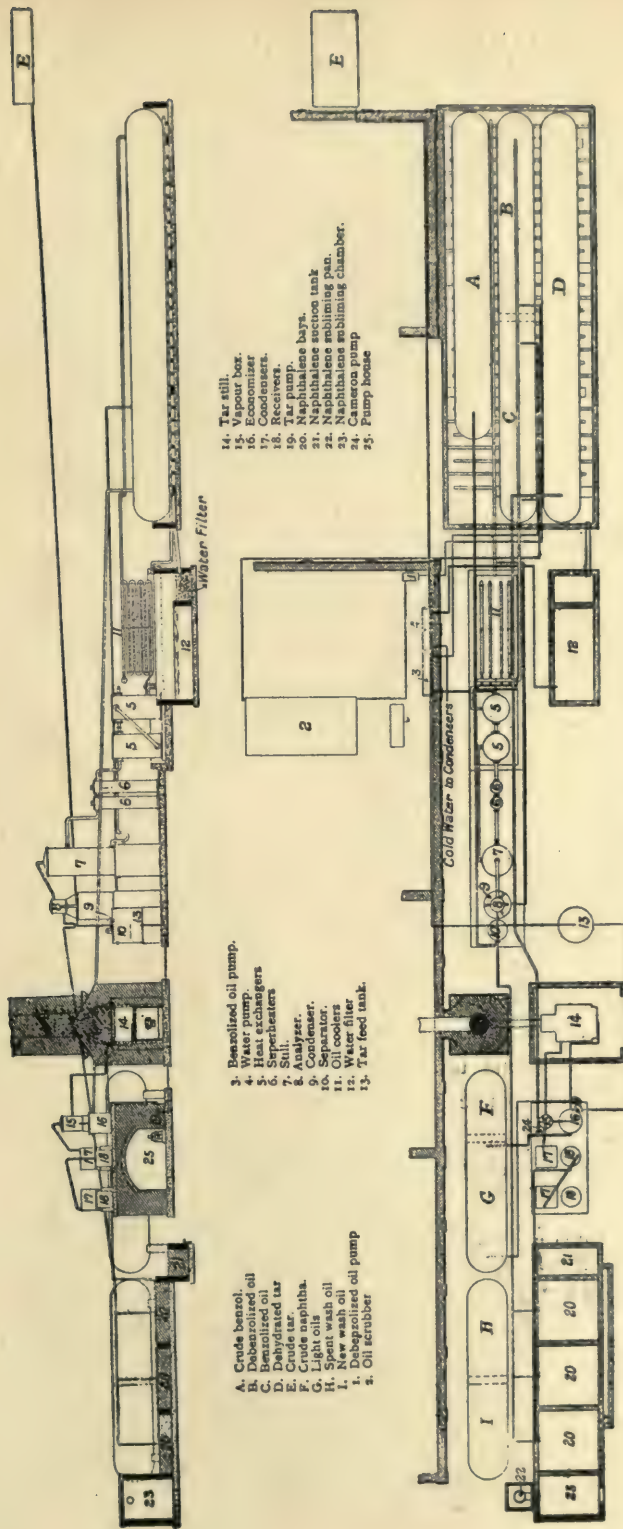


FIG. 179.—BENZOL AND NAPHTHALENE RECOVERY PLANT AT THE ETEURIA GAS WORKS.

of the superheaters near the tops, where the hot oil from the preheaters came into contact with the iron casting heated from the inside with live steam. This corrosion may be due to the action of either cyanogen compounds or to ammonium chloride, but this has not yet been fully determined. It must be remembered that the oil scrubber is placed at the inlet to the purifiers, as it could not be conveniently arranged at the outlet. Had it been so placed, it is probable that this difficulty would not have arisen. The remedy adopted was that of fixing an outer cast-iron jacket to the steam-chamber, the intervening space being run with molten lead. Wooden strips were also bolted to the ribs of the steam-chamber, making a more or less tight joint with the outer casing. This prevented short-circuiting, and ensured a more rapid circulation of the oil through the apparatus. The steam pressure available was 80 lbs. per square inch. It was formerly thought that the higher the pressure, the the greater would be the heat in the still; but the steam consumption had necessarily to be cut down to the lowest limit in order to keep down the water content of the oil, and so prevent emulsification. The result was that the sensible heat of the steam was so diminished by its expansion through a small orifice that the heat of the oil in the still was actually lower than that leaving the last superheater and entering the still. Under these conditions it was only possible to make 50 to 56% benzol, and much trouble was experienced through the emulsification of the oil, which had to be continually replaced. A gas-fired superheater was then fitted on the steam supply to the still, which raised the temperature from 104° to 118° C., and effected a great improvement in working for some time. But owing to its irregular action it had eventually to be discarded. About the same time it was deemed advisable to instal a plant for the dehydration of tar, and in the spring of 1917 an order was placed with the Chemical Engineering Company, who were the contractors for the benzol plant. The dehydrating plant was so designed that it should be capable of producing wash-oil from tar as well as treating the spent wash-oil for its rectification. It was, therefore, fitted with two condensers in series. The still is of the well-known cast-iron coil type set in brickwork—No. 14 on the plan (Fig. 179). The plant when first started-up was used for wash-oil rectification, the temperature of the still being kept at 200° C., and the still pressure at 50 lbs. per square inch. The results were quite satisfactory at first, but trouble was soon experienced through the coil becoming over-heated—presumably due to the irregular working of the pump on the thick emulsified oil. It was subsequently found more

convenient to treat a proportion of the wash-oil while the plant was being used for tar dehydration. In this way all the trouble was overcome, and the plant was found to be capable of working up to a temperature of 230° C., at a pressure of 50 lbs. per square inch—thus producing a satisfactory proportion of middle oils, eminently suitable for benzol extraction. The naphtha which is not suitable for mixing with the benzol is run into tank F, and sold as crude tar naphtha; the light oils being run into tank G. The heavier fractions are run into one of the tanks No. 20, where they are allowed to cool for the

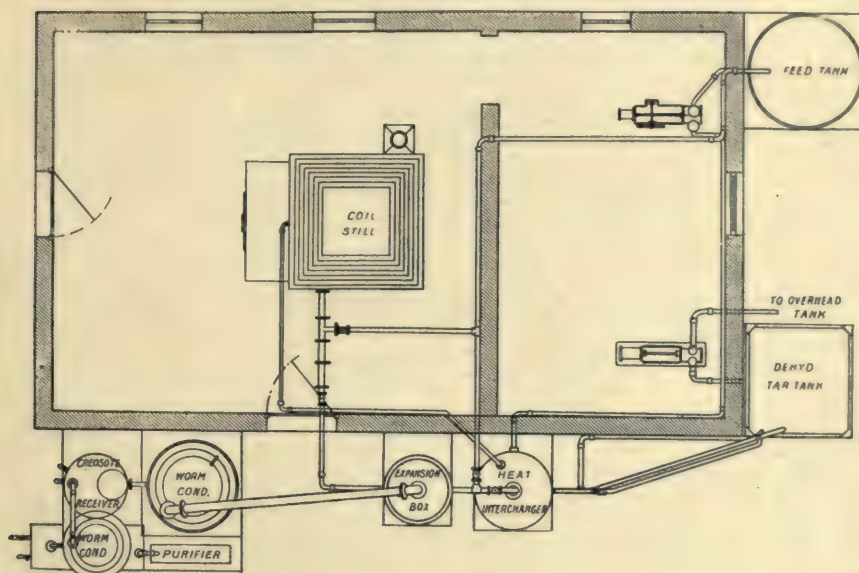


FIG. 180.—PLANT AT THE MAIDSTONE GAS WORKS FOR THE PARTIAL DISTILLATION OF TAR IN A WILTON CONTINUOUS STILL.

purpose of separating the naphthalene. Each naphthalene tank is fitted with a perforated iron plate, and each hole is fitted with a wooden plug. By removing these plugs one by one, the water and various gravities of oil are allowed to run into suction tank No. 21 and thence pumped to where they may be required. It will be noticed that both the benzol and tar plants are arranged at such levels that gravitation may be utilised to the fullest extent; but pump No. 24 is so fitted that the contents of any one tank may be transferred to any other tank. The tanks are divided by partitions, the contents of each being approximately proportioned to the quantities of each product obtained.

The partitions of the tanks, and the settling tanks, are built of wire-cut blue bricks set in cement, and washed with cement and Ironite in equal parts. The latter composition has been found capable of resisting the action of any of the products and preventing leakage."

The Wilton Continuous Still.—Fig. 180 shows the Wilton plant for the dehydration of tar at the Maidstone Gas Works. The plant was described by Mr. Frank Livesey, B.A., Assoc.M.Inst. C.E., in a paper read before the Southern Gas Association, on "Some Notes on the Partial Distillation of Tar in a Wilton Continuous Still." The plant has a capacity of 4,000 gallons of crude tar per 24 hours, and consists of a feed tank of 550 gallons capacity, heat interchanger, cast-iron coil-still, consisting of twelve circular coils 3 feet diameter and 2 inch bore expansion box, from which the tar flows to the heat exchanger, and thence to the finished tar tank. From the expansion box the vapours pass through a cast-iron worm condenser, and thence to the light oil receiver. The various vessels are arranged as shown in the illustration. The still is placed in a brick and tiled building to protect it from the weather, and also to shelter the man engaged in operating the plant. At night time this is important, for since the operator can get his meals in comfort, he has no temptation to leave the plant. At the far end of the building a space is shut off by a wall for the two pumps. One of the pumps is used to force the tar from the feed tank through the plant, and the operator, when firing-up or sitting down to his meals, can see the fly-wheel of the feed pump. It is thus never allowed to stop, as stoppages in time lead to the pitching of the coil. The other pump is for pumping the treated tar from the receiving tank to an overhead boiler for loading purposes. This boiler is of 5,000 gallons capacity, and is fitted with a steam coil for cold weather. The division wall was provided to protect the pump in case of fire, and to enable the operator to safely get to the pumps and shut off the tar supply. The furnace is fitted with Wilton's forced draught, and from the back of the furnace a central uptake leads the gases to the top of the coil, whence they pass down the annular space and up the flue. After a short experience of the plant it was found that, when working at a temperature of 130° C. in the tar, it was not possible to extract the water content. This content was high, and some 4 or 5% of water remained. The steam-blast was increased to the maximum which the flues could deal with. It was found that the crude tar was not leaving the heat interchanger at more than 60° C., and so between the heat interchanger and the still a steam-heater was added, consisting of an old oil carburettor. This

brought up the temperature of the tar to 100° C. before entering the coil, and enabled the temperature to be kept up to 150° C. at the outlet of the coil with an output of 3,000 gallons per 24 hours. When the plant was first installed, a 2-inch pipe was laid from the top of the oil-receiving box to the foul gas main at the inlet of the exhaustor—a distance of about 40 feet; but this arrangement was found to be unsatisfactory, owing to the deposition of ammonium salts in the pipe. It was also found that working at 150° the condenser provided was insufficient to cool the vapours sufficiently on entering the vapour catch-box. A small washer, consisting of a cast-iron tank with a perforated dip-pipe running its complete length and sealed about 2 inches in the liquor, was provided, and a vent was furnished from this washer to an oxide purifier. This arrangement worked satisfactorily for some time, and prevented loss of ammonia, and when working up to 150° C., cool liquor could be run off to the well.

The Hird Continuous Tar Distillation Plant.—Fig. 181 shows the Hird patent continuous tar distillation plant as made by Messrs. W. C. Holmes and Co., Ltd., of the Whitestone Iron Works and Turnbridge Foundry, Huddersfield. The illustration is from a photograph of one of these plants erected at a gas works in the North of England, and the plant has a capacity of 20 tons of crude tar per twenty-four hours. Numerous installations of this type of plant are in operation, ranging in size from 20 tons to 100 tons per day. The plant consists of the following apparatus:—(1) three stills of cast-iron, each 9ft. long, 3ft. wide, and 3ft. 6in. deep, through which the tar is passed in series. Stills Nos. 1 and 2 each contain three 6in. internal diameter steel boiler tubes $\frac{1}{4}$ in. thick, expanded into $\frac{1}{2}$ in. steel end plates, which are bolted to the flanges of the cast-iron casing. These tubes form the only medium by which the heat is transmitted to the tar, which is made to flow along the outside surface of the tubes by means of baffle plates. The tubes may be heated direct by means of burners if a supply of coal gas or producer gas is available, or by a coke breeze furnace. Still No. 3 is heated by the hot gases from No. 1 and No. 2, which are passed through a flue beneath it and thence to the chimney. A small quantity of steam is blown through this still by means of perforated pipes. (2). A pitch cooler of cast-iron, of the same dimensions as the still, which also acts as a heat exchanger, being fitted with tubes (similar to Nos. 1 and 2 stills), through which the crude tar is passed. The finished pitch overflows from the cooler without fumes, and is run direct to the pitch beds. (3) Three heaters. The products of dis-

tillation leaving the three stills are passed through cast-iron coils in the heaters, which consist of cast-iron casings 3ft. diameter by 6ft. deep, and through which the crude tar is passed in series before going to the pitch cooler. The heat from the vapours in the coils drives off the water and naphtha, which are collected from the tops of the heaters and passed through a separate coil and condensed. (4) The condensers. The vapours from the stills are finally cooled and condensed in the three condensers, from which the oil passes through sight overflow boxes to the receiving tanks.

From the above it will be seen that the plant produces simultaneously four fractions of distillates, the character of which may be varied at will by varying the temperatures of the stills. Each still has its own heater and condenser and sight overflow, producing (a) naphtha from the heater; (b) light oil from No. 1 still; (c) creosote oil from No. 2 still; and (d) anthracene oil from No. 3 still. The temperature of each still is registered by an electrical pyrometer.

In addition to the above, the following plant is necessary:—*Naphthalene Separators*.—The distillates leaving Nos. 1 and 2 stills both contain naphthalene, and should therefore be cooled and separated from the crystallized naphthalene before storage. For this purpose four D-section pans, each about 7ft. by 3ft. 6in. deep by 22ft. long, of 2,500 gallons capacity, should be provided. *Storage Tanks*.—The extent of storage provided depends on the means available for disposal of the products. There should be at least one tank for each of the four products, capable of holding the equivalent of two railway tank waggons. A convenient size would be 7ft. diameter by 22ft. long; capacity, 5,000 gallons, or 25 tons approximately.

The working of the Hird plant was the subject of a report to the Burnley Town Council by their engineer, Mr. J. P. Leather. The Gas Committee of the Council had recommended that a tar-distilling plant should be installed, and the Engineer reported upon an installation of Hird stills at the works of the Talk-o'-the-Hill Colliery. He said:—“They have there an installation of Hird's patent stills of the same size as those which I suggest for our works. They have, however, four stills in place of three, and pass through the plant 26 tons of tar in 24 hours. The plant is the first one erected of this type. Some slight modifications and improvements are incorporated in newer plants, but no essential difference. The Manager spoke very highly of the ease of working and absence of anxiety, as compared with the older kind of plant, of which he has had a lengthy experience. The most striking features are the small size of plant compared with the amount of work

done, and the remarkable absence of smell and nuisance. I have been on many works, and this last attribute appealed very strongly to me. The plant was in full work. The flow of the various distillates and of the pitch could be plainly seen, but no smell was discernible. I made particular inquiry into the question of wear and tear. The only part on which there is any serious depreciation is in the heating tubes. Those in the second still corrode most quickly, and require renewing after about 7,000 tons of tar have been dealt with. They have had practically no other repairs, the total cost being under $2\frac{1}{2}$ d. per ton of tar. I

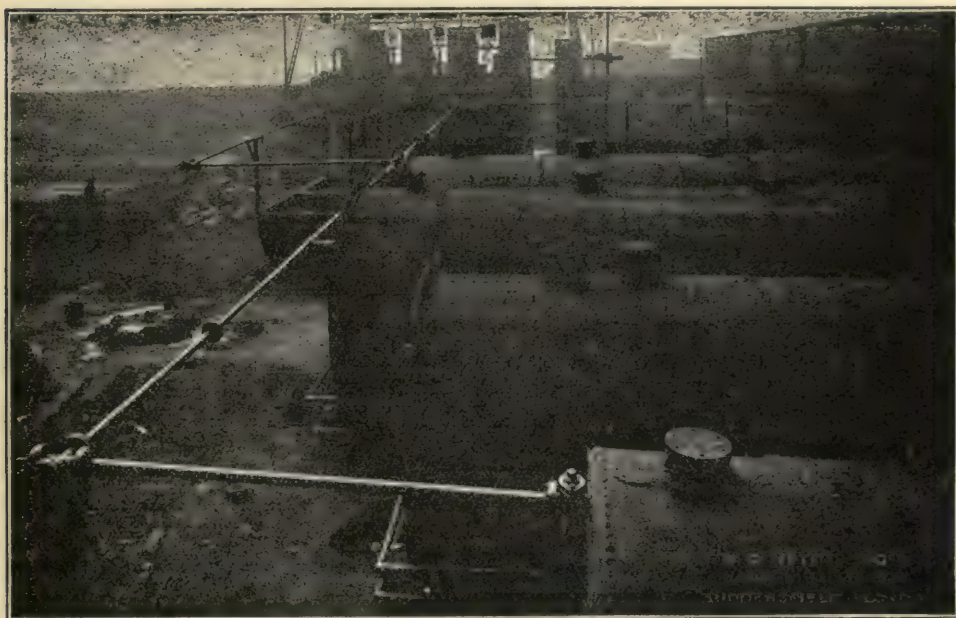


FIG 181.—THE HIRD CONTINUOUS TAR DISTILLATION PLANT.

allow, however, 10% depreciation on the full cost of the plant, which would cover all probable happenings. The first two stills (in a three-still plant) are heated by gas. The third has live steam passed into it, which carries off the vapours of anthracene oil. From this the pitch passes into a pitch-cooler, before running out into the pitch-bays, where it solidifies. Before entering the first still, the tar is partially distilled and dehydrated by passing through heaters which contain coils conveying the hot vapours from the stills to the condensers. In this way the water and crude naphtha are driven off gently without any risk of frothing by heat, which would otherwise be wasted. The

apparatus therefore not only works smoothly but with economy of heat. The plant being kept at a constant temperature, supervision is reduced to a minimum. At Talk they work continuously for seven weeks. The plant is then stopped, and the stills cleaned out, except the last still, which does not need cleaning. The facilities for cleaning have been improved in more recent installations, and I am informed that the operation of cleaning does not now involve more than 24 hours stoppage. The tender of Messrs. W. C. Holmes and Co. is £2,800 for a plant having a capacity of 20 tons of tar per day. This includes five tanks of a capacity of 5,000 gallons each for the products of distillation, and also naphthalene pans of a like capacity for crystallizing out naphthalene from the creosote oil, with the necessary creosote pump and connections. I propose to use as a tar-feed tank an existing cistern formerly employed for the water supply of the works. I have added to the estimate £200 to cover the cost of pipe-line, air-compressor, and other incidental expenses, making the total estimated cost (December, 1916), £3,000. The tar we produce yields the following products per ton:—15 gallons crude naphtha, 28 gallons light oil, 20 gallons creosote, 24 gallons anthracene oil, 11½ cwt. pitch."

The "Southport" Debenzoling Plant.—Fig. 182 shows a debenzoling plant for small gas works carbonizing not less than 3,000 tons per year, described by Mr. John Bond, of Southport, in a communication to the Midland Association of Gas Engineers and Managers. It had been generally considered that a complete benzol extraction plant was only practicable for works carbonizing not less than 20,000 tons a year.

The benzolized oil is continuously pumped into an overhead syphon, which is fitted with an overflow. By the adjustment of a cock marked A on the plan, a regular supply of oil is maintained. The benzolized oil passes through a heat-exchanger, and enters at the bottom of the twin heating tubes at B. Connected to the inlet-pipe there is an extension-pipe to the debenzoling oil cooler, which pipe is fitted with a cock, so that at any time the oil can be drained out of the heating tubes, the end flanges removed, and the tubes cleaned when necessary. The oil passes out of the 16-inch heating tubes by means of two 2-inch pipes, at the top of which are mounted thermometers; these thermometers are fixed into the top plugs, and made tight by cement. The tubes are connected to the chamber C in the middle of the fractionating column. The benzolized oil, which is heated to a temperature of between 110° and 120° C., passes into the lower portion of the fractionating column,

through a quantity of steel turnings and rings, as shown on the plan. At the base of the column there is a small chamber, into which a volume

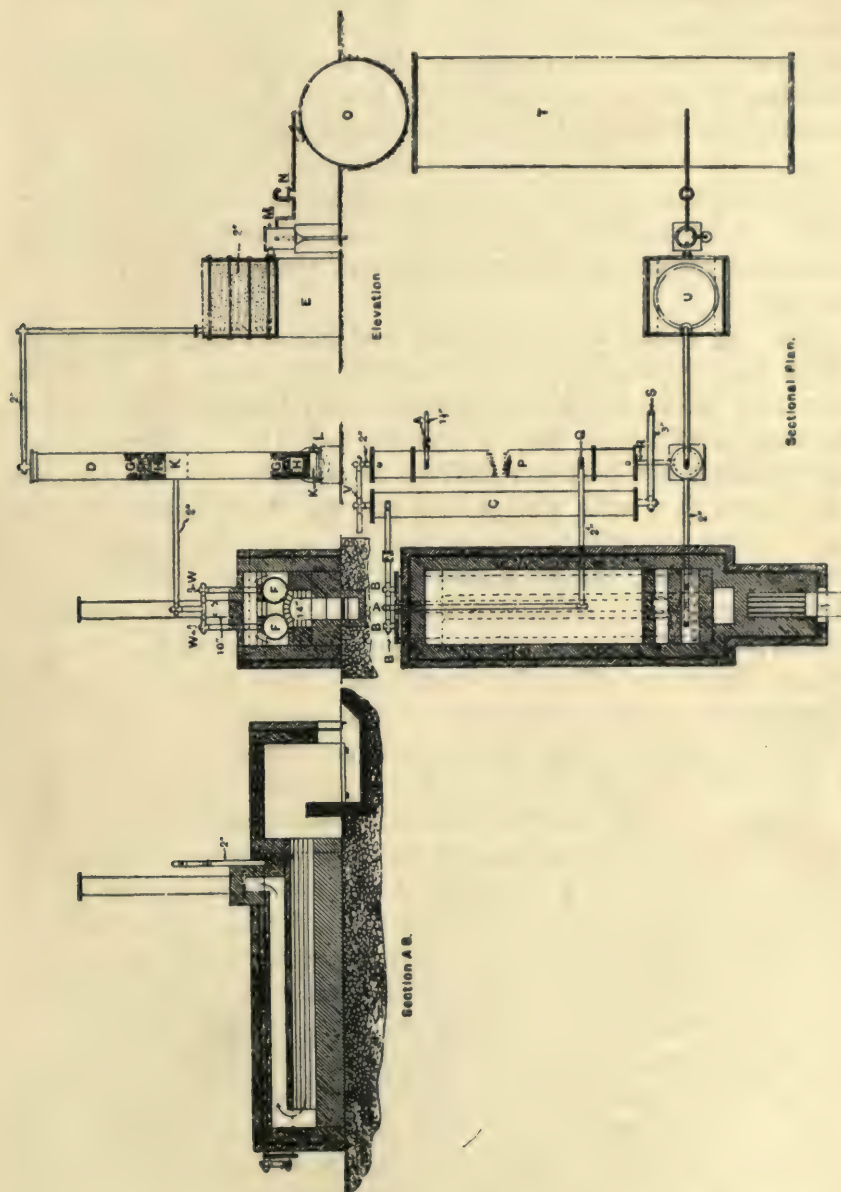


FIG. 182.—THE "SOUTHPORT" DEBENZOLIZING PLANT.

of steam is passed. The steam meets the hot oil as it passes through the mass of turnings and rings. The upper portion of the column is

filled in a similar manner to the lower portion. By a proper adjustment of the steam, a 65% benzol can readily be obtained from the benzolized oil. It is possible to obtain as high as 80% benzol; but if this quality is maintained, a certain amount of benzol returns with the oil to the storage tank. The benzol condenser D is made from 2-inch lead pipe, and attached to a small syphon-pot at the lower end, in which separation of the water from the benzol is effected. In the working of the "Southport" plant, the steam is so regulated that the condensed water coming along with the benzol is equal in volume to the crude benzol. The plant is capable of debenzolizing 250 gallons of benzolized oil per hour, and the maximum amount of 264 gallons of 65% benzol may be recovered in one day.

Fig. 183 shows the oil washing and debenzolising plant at the Gorleston Gas Works, which was described by Mr. E. F. Keable in a paper read before the Eastern Counties Gas Managers' Association. Seven tanks in all are in use. A suitable tank (No. 5) is sunk below ground to receive the tank-cart delivery of creosote oil. The oil from here is pumped to a main storage tank (No 7). From here it is raised into an 80 gallon tank (No. 1) fixed to feed the washer by gravity. This tank contains a float, and by means of a weight passing over a gauge board the regular feed of oil per ton of coal carbonized can be regulated. In order to produce even results this is important. The washer in use is a Kirkham, Hulett and Chandler centrifugal. After removing the last traces of ammonia in the bottom three trays, the top four are fed with creosote oil at the rate of 50 to 60 gallons per ton of coal carbonized. They had found that by adding one tray to the original working—thereby allowing the gas longer time contact—better results were obtained. Frothing over, as at first experienced, had been overcome since an extra tray was added by draining debenzolized oil away from the washer by means of a run-off cock at the bottom of the last oil tray. A thermometer is fitted in the syphon conducting the oil to the washer; and by using one for the gas also differences in temperatures can be watched and water trouble in the oil avoided by keeping the temperature in the oil above that of the gas. During winter months this had been accomplished by feeding-tank No. 1 supplying the washer with warm stripped oil from tank No. 5. By this method oil at the syphon had usually been from 5° to 6° above the gas. After leaving the washer the benzolized oil passes by gravity to tank No. 2. Each day before starting to strip the contents of this tank are pumped to the main benzolized oil storage tank (No. 3). The still is fed from here by a further 80 gallon tank (No. 4). This also has a float, etc., and feeds by gravity. The rate of flow can

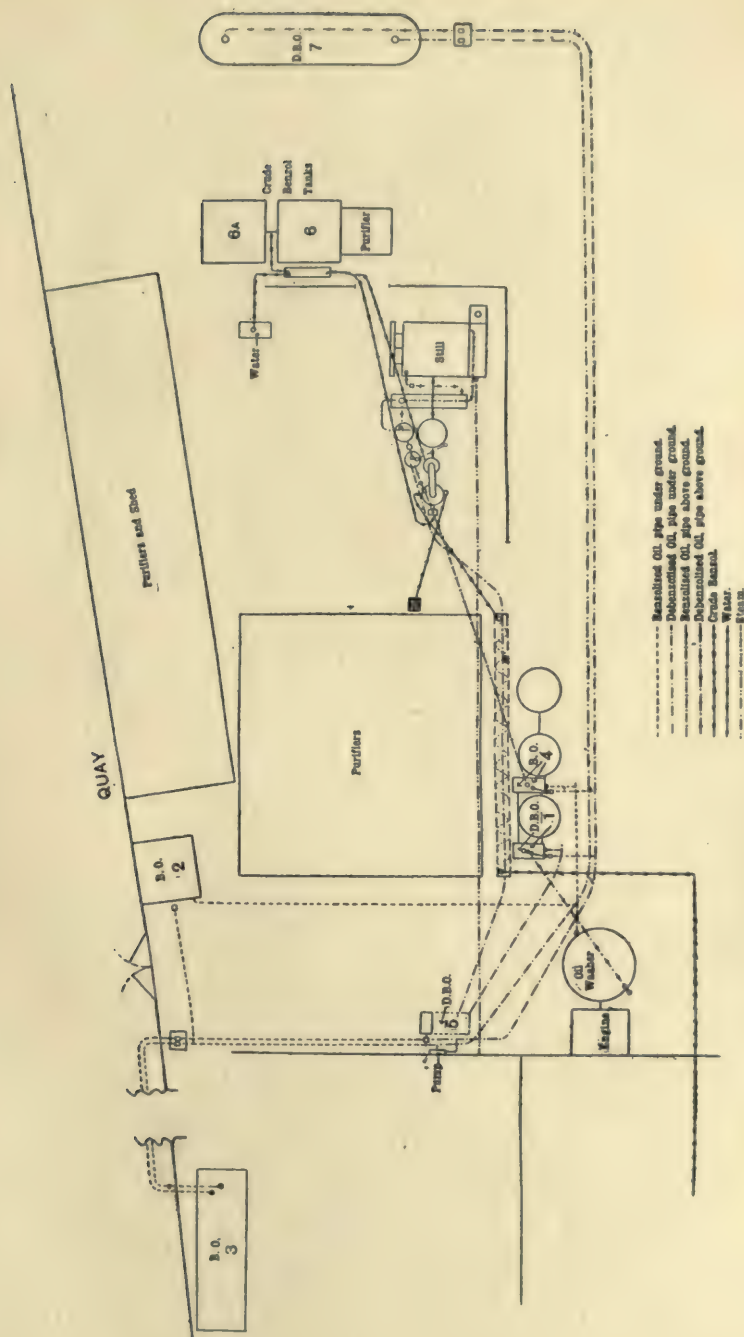


FIG. 183.—OIL WASHING AND DEBENZOLISING PLANT AT THE GORLESTON GAS WORKS.

by this means be both controlled and recorded in a way which is impossible by a pump feed. Regularity at this point was not only desirable but necessary. After passing through the still the hot debenzolised oil is cooled in the following manner: The oil passes through the heat interchanger, and then into the coil of a home-made cooler, and is here reduced in temperature some 30° by the counter flow of the incoming benzolised oil. At the outlet of this cooler it passes through a final water cooler formed by a straight pipe passing through four lengths of 3-inch cast-iron pipe below ground. Water in this is flowing counter to the hot oil. They had an abundant supply of cold well water for cooling purposes. This was necessary, otherwise benzol would be lost to the purifier and the stripped oil insufficiently cooled. The water ultimately falls into a sight funnel and away to waste. The cooled oil then falls by gravity into tank No. 5. From here it is pumped into tank No. 7, having completed its cycle. All the tanks are linked by pipe lines, and the necessary pumping is done by a double-acting wall pump specially made for the purpose by J. Evans and Sons. In order to avoid mistakes, it was decided at the outset that the operator should know the tanks by numbers. Further, all suction valve wheels are painted red and deliveries white.

The distilling plant was originally erected by Hird, Chambers and Hammond for tar dehydration. The alterations and additions to these are as follows: On the outlet of the original condenser there has been fixed a fractionating column, one end of which is sealed in a pot having a suitable overflow, the top end being attached to the water-cooled tubular benzol condenser. On the outlet of this condenser is fixed a sight box having outlet for crude benzol to a tank and at top for waste gases to a small lime purifier. These additional parts were supplied by the original makers of the plant. The more recently erected tar plants made by this firm were at the outset fitted in this way. A supply of live steam had been added to the still. At first two supply points were in use, each having a cap in which two small holes were drilled. These they found soon became blocked, and in place of these one central supply had been fixed in the crown. Steam is caused to pass through the waste gas flue on its way to the still. The steam pipe in the flue passes backwards and forwards three times, finally passing out at the crown, at which point a valve and branch for the steam pressure gauge is fixed. In order further to superheat the steam as much as possible, the three iron flue elbows at the rear of the still are lagged and encased in sheet iron, thereby throwing as much heat as possible into the flue containing the steam pipe. The steam pipe

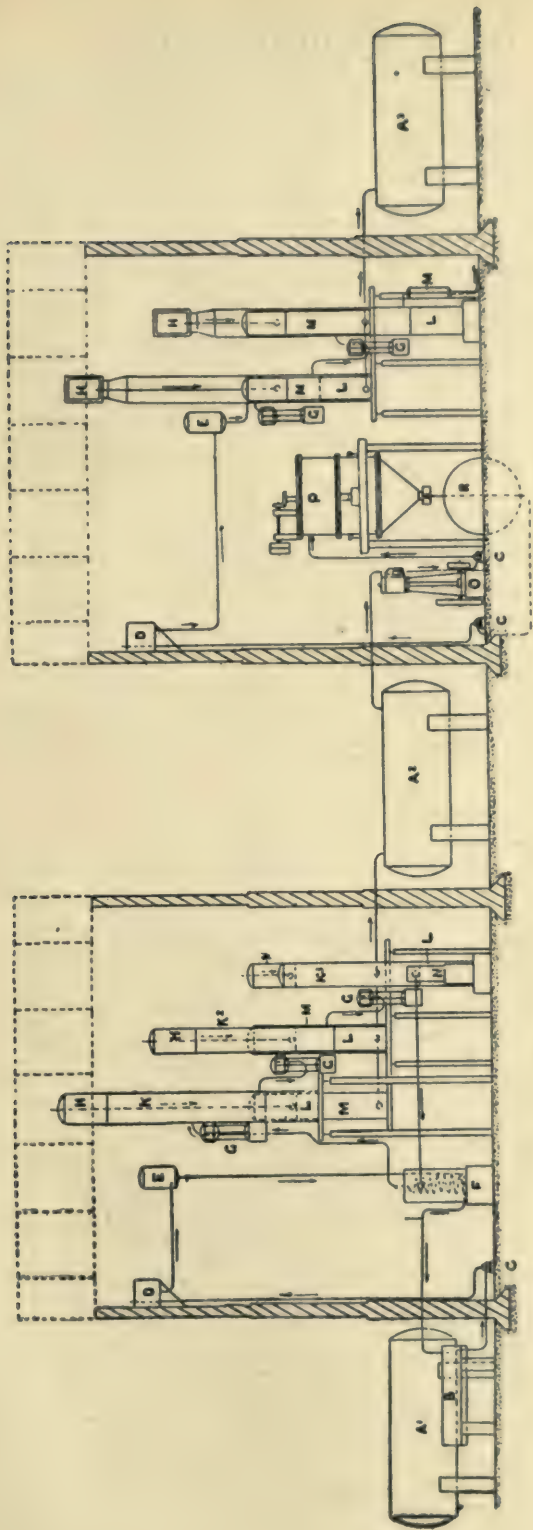
passes through the still lagging and, entering the still at the centre of the crown, reaches to within three inches of the bottom. Arrangements had been made to make its removal an easy matter when a tar run was on.

As the entire plant stands in the open, it was necessary to lag wherever possible. The original or primary condenser is lagged, and thus plays an important part in heating the benzolised oil on its way to the still. As arranged, the incoming benzolised oil is first raised in temperature by benzol vapours passing through the coil of the primary condenser, and finally by the stripped oil passing counter through the heat interchanger. Before entering the still the oil is thus raised in temperature to about 70° C. The entire still is lagged by means of slag wool between the brickwork; and, although the plant is shut down at night, the temperature in the still next morning is usually between 26° and 37° C. When the benzol is distilling, the crude spirit from the condenser and seal pot unite prior to entering the separator, the spirit going forward to tank No. 6 and the water, after being measured, to the liquor well. A sample of liquid taken prior to the separator usually shows half-and-half. The steam used in the still was equal to an evaporation of 10 lbs. of water per gallon of spirit produced. In making the separator, they found it necessary to fix a plate-glass lid in order to avoid loss of benzol by evaporation. This further afforded a ready means of watching the flow. A glass sight hole in the side of the separator was used for keeping the level of the spirit and water constant. Suitable valves are fixed so that when tar distilling is in progress the heavy fractions from the seal pot at the outlet of the primary condenser collect in a separate small tank, the light oils going forward to the separator to be collected in tank No. 6. Thus by collecting separately the value of the plant in this direction will be improved. In order to assist in easily changing over from tar to oil, or *vice versa*, pipe-line connections had been laid in order to empty the plant of its contents while either the tar or the oil is still hot.

The plant was originally made to deal with approximately 600 gallons of tar per day, but for debenzolising wash oil they found it capable of dealing with from 70 to 80 gallons per hour. It had therefore not been necessary to work at night, and they arranged to have the washer feed tank pumped up as required during the night by a stoker. With the still temperature at 129° C., steam pressure up to the controlling valve (60 lbs. per square inch), and oil passing at the rate of from 70 to 80 gallons per hour, they had produced on an average 2 gallons of spirit per ton of coal carbonized, containing on

an average 68% at 120° C. The oil was saturated to 5%, this having been found the best point for their working. The debenzolised oil usually showed only 0.20 to 0.80% at 180° C. The rate of feed to the washer and distilling plant was recorded hourly, as were also the temperature of the still and steam pressure. Samples of spirit, benzolised and debenzolised oil were taken at frequent intervals during the day. These were tested for the quality of the spirit, the percentage saturation of the oil, and the stripping efficiency of the still. They had used from the start a light creosote oil of 1.02 specific gravity (4° Twaddle), low in naphthalene content. Being light, its life was lengthened; and so far their entire parcel had not been changed; but they had at intervals added fresh oil.

Fig. 184 shows a continuous benzol distillation and refinery plant by Messrs. Blair, Campbell and McLean, Ltd., of Govan, Glasgow. The plant is described as follows in the Coking and By-Product Section of the *Gas World*. Referring to the diagram, the crude 65's benzol or any other convenient strength is fed from a storage tank by means of a feed pump to an overhead tank which is provided with special mountings. From here the crude benzol flows, *via* the liquor or benzol regulator, to a preheater, when the outgoing creosote oil gives up most of its heat to ingoing benzol. The preheated crude benzol is next fed into the first fractionating column, provided with a special type of vertical dephlegmator, and, should benzols of varied composition be treated, special arrangements are made to distribute them, at various heights, to the column. The benzol whilst descending the first fractionating column has a particularly stringent washing effect on the ascending vapours, and this has great influence on the fractionation and production of the 90-95's crude product. The benzol flowing from the bottom tray is fed into the heating apparatus, which is arranged on a similar principle to the vacuum heating system of the makers. Here the benzol is evaporated, leaving the heavier residues, which flow into the second column. The amount of steam used in this first benzol column is extremely small, and is regulated by means of an improved type of steam regulator. The residue flowing to the second column undergoes similar treatment, and results in the evaporation and subsequent condensation of 90-95% crude toluol, the solvent naphtha being distilled off into the last column, while the crude creosote oil overflows from the seal pot to the preheater, where it is discharged to the naphthalene pans, ready for salting out the naphthalene. The crude concentrated products are allowed to accumulate until a sufficient quantity is obtained for washing purposes and subsequent fractionation in the refining plant.



- A¹ Storage tank for crude benzol.
- A² Feed tank from condensers to washers.
- A³ Storage tank for finished products.
- B Naphthalene pans.
- C Feed pumps.
- D Overhead tanks.
- E Regulators.
- F Preheater.
- G Steam regulators.

- H Dephlegmators.
- K Crude still, No. 1.
- K² Crude still, No. 2.
- K³ Crude still, No. 3.
- L Liquor heaters.
- M Condensers.
- P Washers.
- R Run-off from washer and feed to refining stills.

FIG. 184.—CONTINUOUS BENZOL DISTILLATION AND REFINERY PLANT BY BLAIR, CAMPBELL AND McLEAN, LTD.

The whole of the stills are in the same building and arranged to take up a minimum amount of floor space. The crude concentration products are fed to the washer by means of a small feed pump, and are here treated with the necessary sulphuric acid, water and caustic soda, which are fed from the storage eggs to overhead feed tanks. After the washing is finished the product is run to an underground feed tank for the refining stills. The pump is connected to this tank and feeds continuously to the overhead tank, which distributes the ingoing washed products *via* the liquor regulator to still No. 1. In the case of treating washed benzol the forerunnings are taken off in this first column and efficient fractionation is effected by a special type of horizontal tubular dephlegmator, which is claimed to be a great improvement over the German type of horizontal tubular apparatus, and has special facilities for cleaning the tubes, distributing water and taking away the condensate. The arrangements are not attended by the usual choking by means of naphthalene, which is stated to be always found in connection with the German type of plant. The residue from the first refining column is fed into No. 2 column, where the refined benzol is distilled off, leaving a small amount of toluol residue, which is cooled by means of water, and fed to a small receiver which is directly connected to the feed pump, and can, therefore, be pumped along with the next charge to the refining stills. The refined products flow into their various storage tanks, and are then ready for discharging into railway wagons or drums. The floor space taken up by this type of plant is approximately one-half that taken up by the intermittent type, and the feed and steam control is entirely automatic, being dependent on the existing column pressures; and as these are constant for a given make of product, it naturally follows that very little attention is necessary, and the working proceeds quite smoothly.

Fig. 185 shows the plant designed by Dr. W. B. Davidson and Captain Hilton, for the recovery of toluol at the Nechells Station of the Birmingham Gas Department, and described as follows by the *Journal of Gas Lighting*.

The stream of cooled debenzolised oil coming from the crude still is mixed with a small stream of benzol in tank T_1 . The content of benzol in the mixture is regulated according to the flow of oil and the degree of saturation obtained in the scrubbing process. This will probably be 4 or 5% by volume. The wash oil thus prepared is pumped at the required rate (about 1,300 gallons per hour) by pump P_1 to the top of scrubbers S_2 ; then by pump P_2 to the top of S_3 ; and finally by pump P_3 from the bottom of S_2 to the top of S_1 . The oil now benzolised runs

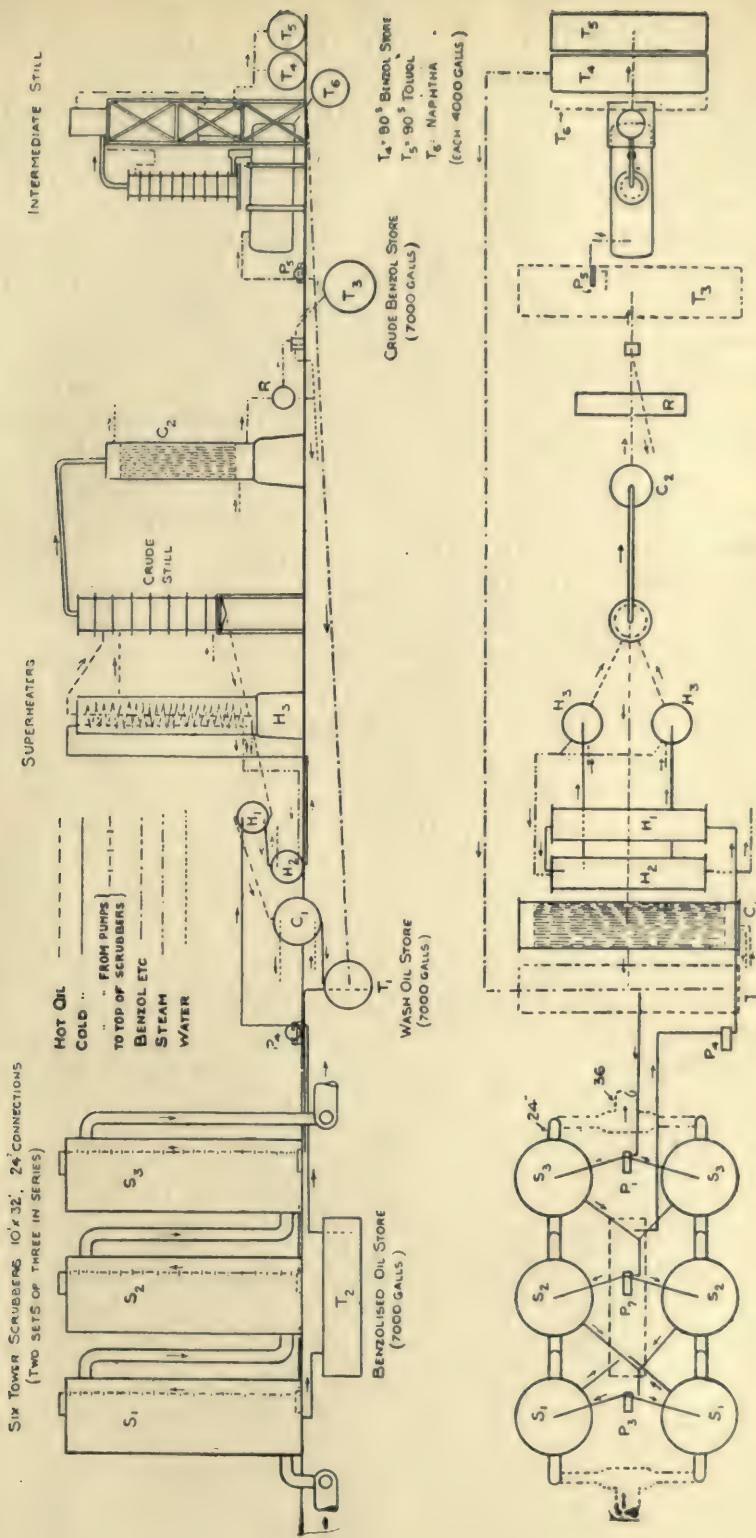


FIG. 185.—TOLUOL RECOVERY PLANT AT THE NICHELLS GAS WORKS, BIRMINGHAM.

from the bottom of S_1 to the store-tank T_2 , whence it is pumped by P_4 in a continuous stream at the rate of about 1,300 gallons an hour through the heater H_1 , where the heat interchange takes place between the cold oil and the hot oil leaving the crude still; then through the exhaust steam heater H_2 ; then through the superheater H_3 (containing a closed high-pressure steam-coil) to the crude still, where it falls down from tray to tray to the lowest compartment—meeting a current of high temperature steam, which deprives it of its crude benzol. The hot debenzolised oil is drawn off—preferably by gravity, and caused to flow first through H_1 , and afterwards through a water condenser C_1 to the store-tank T_1 . The crude benzol vapours and steam are condensed in the water condenser C_2 . The condensate flows to a receiver R , whence it passes through a separator, the crude benzol being stored in the tank T_3 . A charge of about 1,600 gallons of crude benzol is pumped by P_5 daily into the intermediate still (either of the Heckmann or the Savalle type), where it is distilled by steam with the production, firstly, of commercial benzol; secondly, commercial toluol; thirdly, residual zylol and heavier oils. The commercial benzol is mixed with wash oil, together with an additional quantity corresponding to the amount of toluol, etc., permanently removed from the gas. The commercial toluol is re-distilled in a second still of the Heckmann or Savalle type, on a central works, for the production of pure toluol. If the wash oil contains much naphthalene, it may be necessary to return xylol to the gas, to prevent naphthalene stoppages in the district services. Gas oil will probably prove the most suitable oil for washing purposes, as it is generally ready to hand, contains no naphthalene, and readily absorbs crude benzol. The oil may be worked up to a high degree of saturation without appreciable loss of toluene; a considerable proportion of the benzene, which is more difficult to catch, going forward in the gas. The plant is capable of dealing with 800 tons of coal or 10 million cubic feet of gas per day.

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